A Laboratory History of Narcotics

Vol. 1: Amphetamines and Derivatives

A comprehensive laboratory manual discussing the preparation of amphetamines, psychedelic amphetamines, and derivatives

A Book by Jared Ledgard

A LABORATORY HISTORY OF NARCOTICS, VOL. 1: AMPHETAMINES AND DERIVATIVES

A BOOK BY JARED LEDGARD

A Laboratory History of Narcotics, volume 1: Amphetamines and Derivatives®

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A Laboratory History of Narcotics, Vol, 1: Amphetamines and Derivatives

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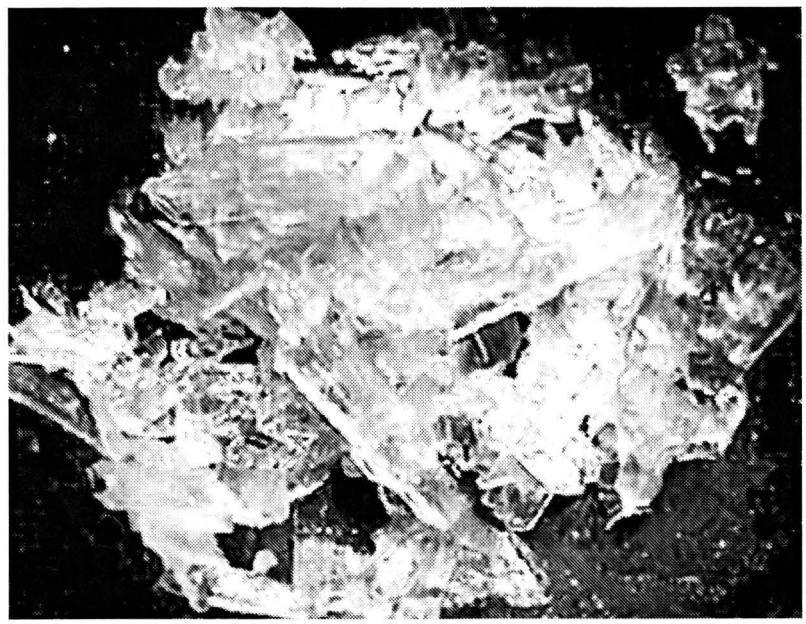
A LABORATORY HISTORY OF NARCOTICS

VOLUME 1

AMPHETAMINES AND DERIVATIVES

A LABORATORY MANUAL

By: Jared B. Ledgard



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SECTION 1: INTRODUCTION

A quick lesson in chemistry

Part 1: Introduction to chemistry

A Laboratory History of Narcotics Vol 1 has been written to teach the art of pharmaceutical sciences to the reader. To do this, you should take a quick, yet vital lesson in chemistry. First of all, the world of chemistry is a fascinating world filled with a huge variety of chemicals, chemical reactions, formulas, laboratory apparatus, and an arsenal of equipment. All these elements are combined and used thoroughly to bring about chemical change of matter from one form to the next. In this book, the form of change that we will deal with mostly, is the formation of compounds that posse's psychological and physical action upon the body. These compounds that posses characteristic psychological effects upon the body are called narcotics.

The world of narcotics is absolutely huge, and in essence, deals with virtually millions of chemical compounds. Amphetamines and derivatives actually range in the tens of thousands, but listing them all in this book would be impossible. Regardless how many possible drugs there might be, most see drugs or narcotics as something evil or something that is a cancer soar on our societies and our civilizations; however, in factuality drugs and narcotics are as old as life itself, and have been around for millions of years longer then we humans have. Narcotics are not evil, nor are they a pain in the side of our society—they are chemical compounds like everything else that exist, not for us, but because of atoms and molecules. These atoms and molecules, and their chemistry, will be around long after were gone, and then some. The chemistry of these substance may be millions of years old, but their chemistry, as it pertains to our benefit, is only a hundred or so years old. Even though ancient civilizations, and numerous cultures have used narcotics for thousands of years, their exact chemical make-up has only been unraveled in the last 100 years—many psychedelic amphetamines only in the last 50 years.

For most of you, the procedures in this book will not make sense at first, or will appear to be complicated; as a result, many of the procedures in this book may seem foreign, or unfamiliar—if this is the case, then at this exact moment, you are in the right place. Bye the time you have read this book, these "foreign" procedures will no longer be foreign to you, but in the meantime, lets get started on the world of chemistry.

The world of chemistry involves every single aspect, corner, and micro drop of everything that is matter. Our solar system and the entire universe all function on a chemical level—In essence, chemistry is everything. The universe and everything in it is composed of atoms and molecules, and within this massive space, there exists tens of millions of chemical compounds—either known or unknown. The compounds that are known make up only 5% of the naturally occurring compounds, leaving a massive 95% of them being synthetic (prepared in the lab)—95% of all narcotics are synthetic. Note: synthetic does not denote anything that is less superior to natural. Synthetic means creating natural in an un-natural way.

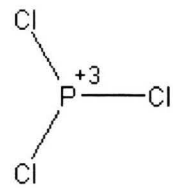
Chemistry has been divided into three fields over the last 100 years to better organize and format the system. The three major branches of chemistry include: Inorganic chemistry, Organic chemistry, and Biochemistry. In short, inorganic chemistry deals with ionic compounds, which make up the chemical compounds that do not contain active carbon. Organic chemistry is the largest branch of chemistry and it deals with covalent compounds, which make-up our everyday items like plastics, drugs, dyes, pesticides, insecticides, resins, fibers, and explosives. Organic means "carbon bearing" which means any compound that bears carbon is classified as organic. Gasoline, turpentine, and candle wax are specific examples of organic compounds. Last but not least, biochemistry studies the field of enzymes, organisms, plants, and animals and their active chemical processes. Genetics research studies the DNA and RNA of living things and is a sublevel of biochemistry. DNA and RNA is composed of organic compounds all linked and actively working together. Biochemistry deals heavily with peptides, amino acids, carbohydrates, ect., ect., all of which play a major role in natural process such as cells, metabolism, and the like.

1. Chemical bonding: Oxidation states

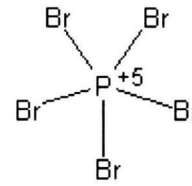
First things first, you need to understand the nature of elements, and their oxidation states (number of bonds). Every single element is capable of forming chemical bonds with other elements (with the exception of a few "noble gases"). The oxidation states are what determines how many bonds a particular element can form, and to what other elements. When elements combine, they form chemical compounds. All of the atoms within a chemical compound show specific oxidation states. Oxidation states are not really states, but definitions of bonding, which are dictated by each individual element. Each element can form any where from either 0 to 7 bonds. These numbers represent the number of bonds the element can form (look at a modern periodic table, such that included in the "Merck Index"—the oxidations states are written in the upper left corner of each element). These numbers clearly indicate the number of bonds each element is capable of forming.

As most people are aware, periodic tables include rows and columns filled with elements. The elements within any given column have similar properties and characteristics along with similar oxidation states. For example, the elements of column 5A on the periodic table include nitrogen, phosphorus, arsenic, antimony, and bismuth. All these elements have similar oxidation

states and properties. Phosphorus for example, can form compounds with three bonds or five bonds (indicated by the numbers +3, –3, and +5). Phosphorus, like arsenic and antimony have oxidation states of +3, –3, and +5. Phosphorus can form either +3 or +5 oxidation states when it bonds to elements with higher electro negativities (also listed on some periodic tables), and –3 oxidation states with elements that have lower electro negativities. Each element has different electronegative energies. Metals for example, have electro negativities ranging from 0.60 to 1.9. Non-metals have electro negativities ranging from 1.9 to 4.0. In essence, elements that are metals combine with the elements called non-metals forming positive oxidation states, with the so-called non-metals forming negative oxidation states.
In a specific example, when phosphorus reacts with non-metals it forms +3 and +5 oxidation states because its electronegative energy is less then the other non-metals, but when it bonds to metals, its oxidation state is –3 because its own electro negative energy is greater then most metals.
Either way, when two elements combine for example, the element with the greater electronegative energy forms negative oxidation states, and the element with the lower electronegative energy forms positive oxidation states. In another example, chlorine and bromine both have greater electronegative energies, so when they combine with phosphorus, the phosphorus forms +3 and +5 oxidation states (see the illustration below). When elements combine they form compounds, which are called molecules.

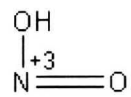


Phosphorus trichloride

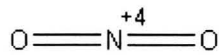


Phosphorus pentabromide

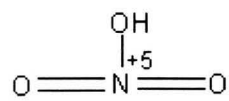
Elements such as lithium, sodium, and potassium form only one bond, because they have only a +1 oxidation state, and because their electronegative energies are quite low (ranging from 1.0 to 0.6). A more complex array of oxidation states is demonstrated in the element nitrogen (a key element found in all amphetamines). It's capable of forming +1, +2, +3, +4, +5, –1, –2, and –3 oxidation states (see the illustration below). Another crucial element, carbon, is capable of forming +2, +4, and –4 oxidation states, and the all important oxygen, forms only a –2 oxidation state. Hydrogen can form +1 and –1 oxidation states. Remember the elements helium, neon, and argon (called the noble gases) form no oxidation states. Note: The oxidation states of each element (and column of elements on the periodic table) have been determined by trial and error over some 200 years of chemical research and study.



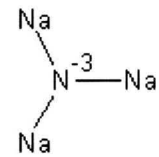
Nitrous acid



Nitrogen dioxide



Nitric acid



Sodium nitride

2. Ionic compounds and ionic bonds

Ionic compounds are composed of elements bonded together that have marked differences in electro negativities. Ionic compounds make up the bulk of "inorganic compounds", and are composed primarily of metals bonded to non-metals. In ionic compounds, the oxidation states of each element follows the same rules governed by the number of bonds each element can form. In the case of ionic compounds, the positive and negative numbers represented by the number of bonds each element can form, is more detailed and also represents a charge attributed to each element. For example, when phosphorus bonds to chlorine, it forms +3 or +5 oxidation states, and the chlorine forms a single –1 oxidation state; however in this example, because the electronegative difference between the phosphorus and the chlorine is not very significant, the resulting phosphorus trichloride or pentachloride is not considered fully to be ionic. However, in the case of sodium chloride, a +1 sodium ion is bonded to a –1 chlorine atom, with each positive and negative mark defined as a charge. Compounds that have their oxidation states defined as actual charges are considered to be ionic. As a reminder, remember that oxidation states (the numbers) define the number of bonds an element can form, nerve mind the positive or negative marks each number has. In ionic compounds the molecules are made up of positive and negatively charged atoms corresponding to their oxidation state number (the number of bonds each element can form, i.e., the oxidation state number defines the number of bonds each element can form, but not their electrical charge in all molecules—just in ionic molecules.
The electrical charge of each element within an ionic molecule is different then the element's electronegative energy. Note: Electronegative energy determines whether the element forms positive or negative oxidation states. Electrical charge is

determined after the atoms combine, and is represented by the positive or negative oxidation state independently from the actual number of bonds each element can form.
As previously stated, chlorine is more electronegative then sodium, so when they combine the chlorine forms a –1 oxidation state (notice on a periodic table that chlorine has an oxidation state of +1, –1, +5, and +7; and sodium has an oxidation state of +1). Some periodic tables give the electronegative energy of each element, and using such a periodic table, you will notice that the electro negativity of chlorine is remarkably higher then that of chlorine. Because the difference between electronegative energies is so great, the chlorine becomes negatively charged, and the sodium becomes positively charged. These charged atoms attract each other, and hence form a bond based on their electrical attractions (like two magnets)—this is the basis of "ionic" bonds.
Oxidation states also determine the number of electrons that can be captured. As previously discussed, ionic compounds like sodium chloride form their bonds based on electrical attractions. These attractions are determined by the number of electrons a particular atom captures. When chlorine combines (reacts) with sodium it forms a –1 oxidation state. Again, because the difference in electronegative energies is so great, the chlorine grabs or captures one of the sodium's electrons. This capturing causes the chlorine to become negatively charged. As a result, the sodium atom becomes positively charged. Atoms become negatively charged when they capture electrons, and become positively charged when they loose electrons. This capturing and loosing of electrons is the scientific foundation to ionic bonding and ionic compounds.
Currently there are about 200,000 ionic compounds known to man (most of them being synthetic). The most common ionic compound is table salt or sodium chloride. Some common examples of ionic compounds include potassium permanganate, sodium azide, sodium nitrate, potassium chloride, sodium fluoride, potassium chlorate, and zinc sulfate. Ionic compounds make up the majority of the earth, solar system, and the universe.

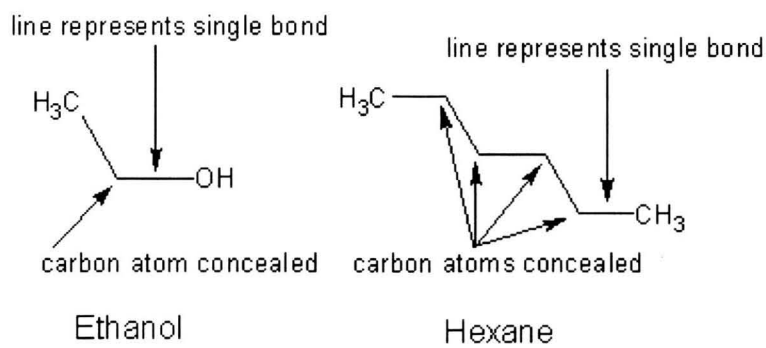
3. Covalent compounds and covalent bonds

Covalent compounds make up the bulk of chemical compounds known to man, but they only a make-up a small percentage of the chemical compounds found on earth and earthly like planets, and virtually most solar systems. As previously stated, there are about 200,000 ionic compounds known to man, with a potential of another 100,000 left undiscovered throughout the universe; however, covalent compounds number in the millions. For example, currently there are 16,000,000 covalent compounds known to man (as of 2003). The possible number of covalent compounds is practically endless, as the combination of these compounds is virtually infinite.
Covalent compounds contain covalently bonded carbon atoms. The term "organic" means 'carbon bearing covalent substance'. Covalent compounds all contain specific carbon atoms, which make-up the foundation or infrastructure of all organic compounds. A covalent compound such as hexane for example, is composed of covalently bonded carbon atoms all bonded together to form a chain—this chain represents the backbone or infrastructure of the molecule. The carbon atoms that make up these backbones or infrastructures, are themselves bonded directly to other atoms such as hydrogen, oxygen, nitrogen, sulfur, phosphorus, arsenic, ect., ect. Such examples of covalent compounds (organic compounds) include: ethyl alcohol, isopropyl nitrate, aspirin, acetaminophen, cocaine, and octane.
Covalent bonds are much different then ionic bonds, as they share electrons rather then "capture" them. Remember that ionic bonds are formed when two or more elements with distinctive differences in electro negativities react with one another—whereby the greater electronegative element captures an electron (or more) from the less electronegative element(s). Covalent bonds, however, are formed when two or more elements combine and the electrons are shared (paired) rather then captured. In order for a covalent bond to form, the electronegative differences between the elements cannot be very significant, meaning their differences are much less then those encountered with ionic bonds.
Covalent bonds cover a whole echelon of reactions, many of which can be very complex and/or require special conditions depending on the chemicals and reaction conditions, and usually require multiple reactions and steps to achieve desired products. In other words, ionic compounds tend to be rather simplified compounds with easy formulas, whereas organic compounds can be huge molecules, which require many steps for their preparation. These multiple steps are the basis for organic chemistry, as it deals with a whole multitude of reactions and functional groups—most of these reactions and functional groups will not be discussed in this book (as it would take about 100,000+ pages), but what functional group reactions that will be discussed are the amino functional groups commonly found in amphetamines and derivatives.
In general, covalent bonds are less stable then ionic bonds. Most ionic compounds are stable solids with relatively high melting points (ranging from 200 to 2400 Celsius). Many ionic compounds can be heated to very high temperatures without any significant decomposition, such examples include: aluminum oxide, iron oxide, sodium chloride, and magnesium chloride. Most organic compounds decompose when heated to temperatures above 300 to 500 Celsius. The high melting points of ionic compounds are due primarily to crystal structure, and the result of strong electrical attractions between the elements and the molecules—these attractions can lead to super strong crystal lattices, as seen in some compounds like aluminum oxide (emeralds), and other ionic oxides (gems and sapphires). There is one mere example of an organic compound that should be demonstrated here; diamonds are composed of covalently bonded carbons atoms, with the molecules forming super strong crystal lattices.
Other then this isolated example, most covalent compounds are solids or liquids with relatively low melting points and boiling points. This is the result of weaker electrical attractions between the molecules. In covalent compounds the weaker attractions

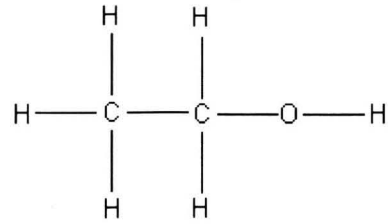
exist primarily because the covalent molecules lack ionic charges, and are thereby not attracted or repelled to each other very much. Because of the lack of electrical attractions between covalent molecules, the boiling points of covalent molecules are the result of "intermolecular" forces (the melting points will be discussed shortly). Intermolecular forces are forces that exist between elements within one molecule upon different elements within another molecule. Such an example would be water, common hydrogen oxide. Water which is composed of two hydrogens bonded to a single oxygen has a significant boiling point of 100 Celsius at sea level, although it is a relatively small and light molecule. The reason water has such a high boiling point for its small size and weight, is due to intermolecular force attractions between the central oxygen atom of one molecule upon the two hydrogens of another water molecule (adjacent water molecule). The non-bonding type attractions (intermolecular forces) that water molecules have to each other is what defines water's boiling point. In another example, methylene chloride (a common solvent you will find in this manual) has a very low boiling point for its size and weight (compared to water). The reason methylene chloride has a boiling point of about 60 degrees less then water is due to even weaker attractions between the methylene chloride molecules to each other. In essence, the weak intermolecular forces between the two chlorine atoms of one molecule upon the two hydrogen atoms of another, is what determines the low boiling point of methylene chloride.
As previously stated, the melting points of ionic compounds are high because of strong electrical attractions between the elements and molecules, but a whole different scenario determines the melting points of covalent compounds. Because solid covalent compounds don't really show any significant intermolecular forces, the melting points of covalent compounds are determined by the shape, size, and bonding angles of the elements within the molecules. For example, think about blocks of wood of the same size, verses wood circular shapes of the same size—which would be easer to stack? Obviously the wood blocks would be much easier to stack then a pile of circular wood blocks. This is basically the essence behind the melting points of covalent compounds—although it gets a little bit more technical then this, but this info will be omitted because it is only of a concern to scientists. Molecules that are shaped properly, will pack together (not literally) much better then molecules that have awkward shapes. Molecules that pack together better, and more evenly, have much higher melting points then molecules that don't pack or fit together very well. Another factor that plays a role in melting point is size and weight of the molecules. Naturally, larger weight molecules tend to have higher melting points and boiling points then smaller weight molecules.

4. Understanding chemical structures and formulas

Understanding molecular structures and formulas is not necessarily needed for this manual (as all procedures are giving with exact quantities) nevertheless, understanding formulas and the like can seriously help you better acknowledge what is taking place during a chemical reaction. Molecular formulas and structures are written using a variety of simple techniques. The most common of these techniques utilizes short lines, which indicate the bonds—of coarse the letters in the illustrations clearly indicate the elements. In short, the lines represent the chemical bonds either ionic or covalent, and the letters represent the elements (see a periodic table for each letter). In this manual, some of the letters have been omitted to reduce drawing time of the structures, and this method of omission is quite common in chemistry literature. In a common example, ethanol and hexane are both written with their central carbon atoms (and hydrogen atoms) concealed. Note: only carbon and hydrogen are commonly concealed in any given illustration. To know when a carbon has been concealed, simply look at how the lines change angles. Because carbon forms four bonds, it naturally contains two hydrogens per carbon (with the exception of alkenes, alkynes, benzenes and phenyls) within the central structure—these hydrogens are also concealed.

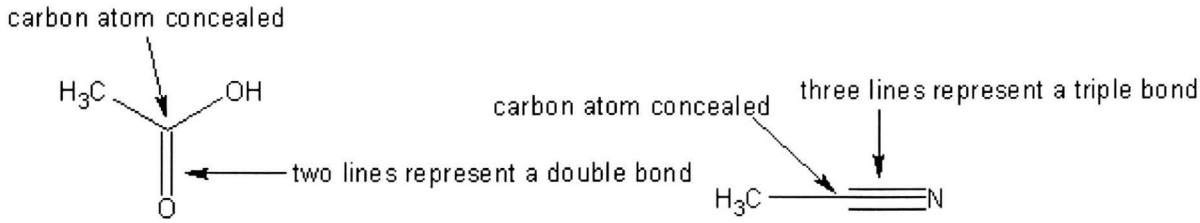


For review, the single lines represent single bonds, and the letters represent atoms. Therefore the letter C represents carbon, the letter O represents oxygen, and the letter H represents hydrogen. In the above illustration the central carbon atom in ethanol is concealed, along with two hydrogens bonded to it—this is the same scenario for hexane with a total of four carbon atoms concealed, along with eight hydrogens. Another method of writing structures and formulas is to use "expanded notation". For example, the structure of ethanol could be written as follows:



Ethanol

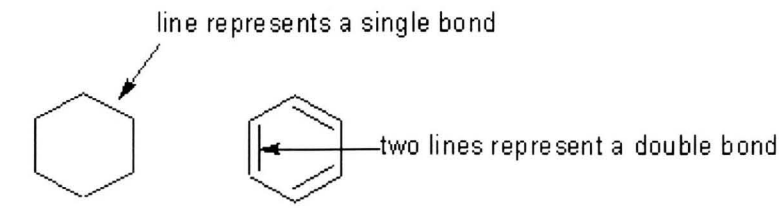
The above illustration is a common example of a molecular structure written in expanded notation. Expanded notation shows all elements within the structure. Expanded notation is seldom used in chemical literature to save writing time. In the following illustration we see a similar written structure with the central carbon atom concealed, along with the corresponding hydrogen. In this example, two lines are written to represent a double bond, in this case between the central carbon and an adjacent oxygen atom. In the right structure, a straight-line triple bond is shown, with the central carbon atom concealed as usual—as suspected, the letter N represents nitrogen.



Acetic acid

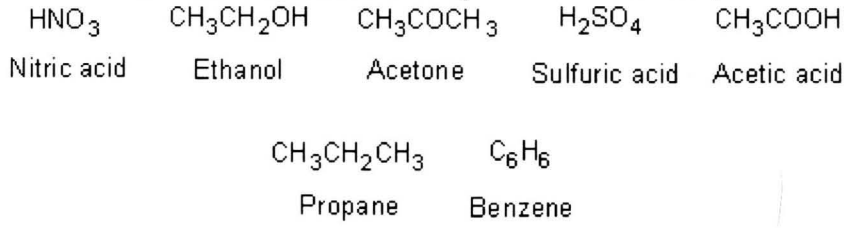
Acetonitrile

Many covalent compounds are composed of rings. Rings are structures with a high degree of stability and belong to either a saturated group, or an unsaturated group. In the following illustration, the structure on the left is called cyclohexane, which represents a saturated ring. The right structure is the classic compound called benzene. In both structures, all carbon atoms have been concealed, along with the adjacent hydrogens—this is how most rings will be illustrated. The benzene structure represents an unsaturated ring. When discussing saturation and unsaturation, rings are not the only covalent compounds capable of these definitions. Many straight chain, and branched structures are capable of forming saturated and unsaturated structures—these are classified as alkynes, alkenes, and alkanes. An example of a unsaturated compound is the chemical acetylene, and an example of a saturated compound is the chemical propane. Another example are oils such as olive oil, which contain long chain unsaturated compounds—mainly oleic acid in this case.



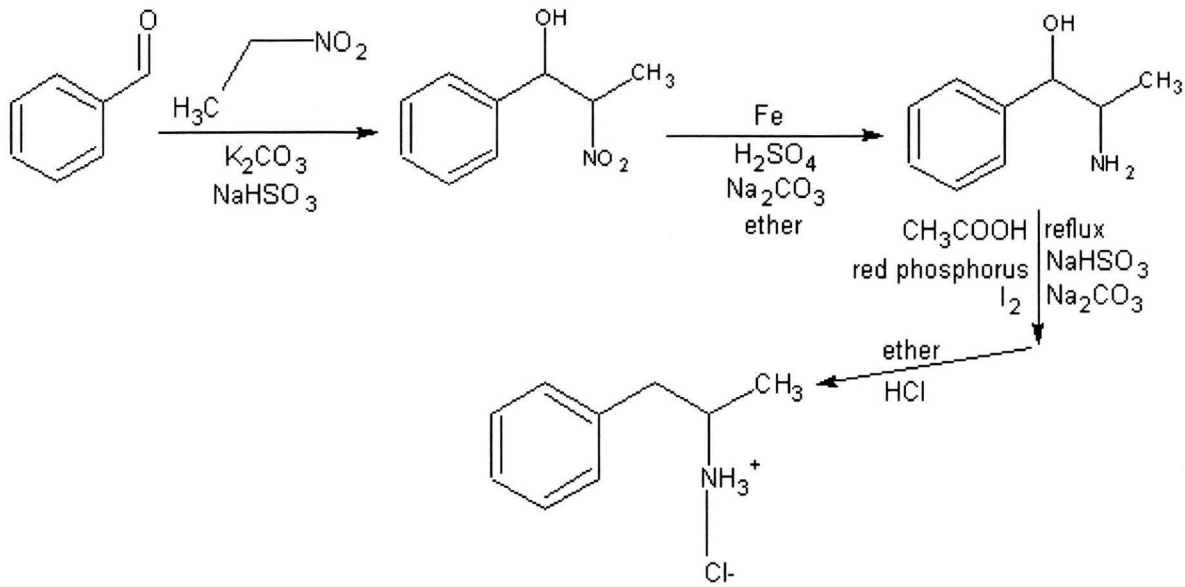
Cyclohexane Benzene

The final, and most common method of writing structures and formulas involves "condensed formula notation". Condensed formula notation simply excludes the lines. To save time and space, many chemists use condensed formula notation. In this book, many of the reagents and solvents will be written in condensed formula notation. The following illustration gives a few examples of condensed formula notation.



5. Chemical reactions

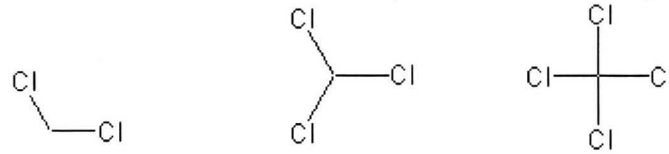
"Chemical reaction equations" are commonly used to illustrate a chemical reaction. In a chemical reaction equation the arrow represents the path the reaction takes. The items listed above and below the arrow represent the reagents, temperature, and/or conditions that exist for and during the reaction. In the following illustration we start with the "intermediate" compound called benzaldehyde (the far left structure). The intermediate compound is usually written on the left hand side, but can be written on the right hand side as long the arrow is pointing to the left. The intermediate is other wise called "the starting compound". In the illustrated chemical reaction equation below, the arrow pointing to the right tells us that benzaldehyde is treated with a mixture of nitroethane and potassium carbonate in the presence of sodium bisulfite. The nitroethane, potassium carbonate, and sodium bisulfite are commonly called the "reagents", and are usually written in condensed formula notation. The reagents are usually written above and/or below the arrow (basic chemistry classes often put the reagents after a + sign, but in the professional world, we don't use + signs). Under most conditions, to shorten the illustration, we omit the by-products formed during the reaction (but sometimes it helps the reader understand better what is going on when the by-product are given; however, by-products will not be given in the illustrations of this book). Now, looking at the rest of the equation, we see the resulting product of this first reaction, is a nitro intermediate, and this new intermediate is then reacted with iron in the presence of sulfuric acid, and then so on, and so on.....Although understanding chemical reactions is not fully necessary to properly use this book, a brief understanding will better help you understand what is taking place.



6. Language of chemistry

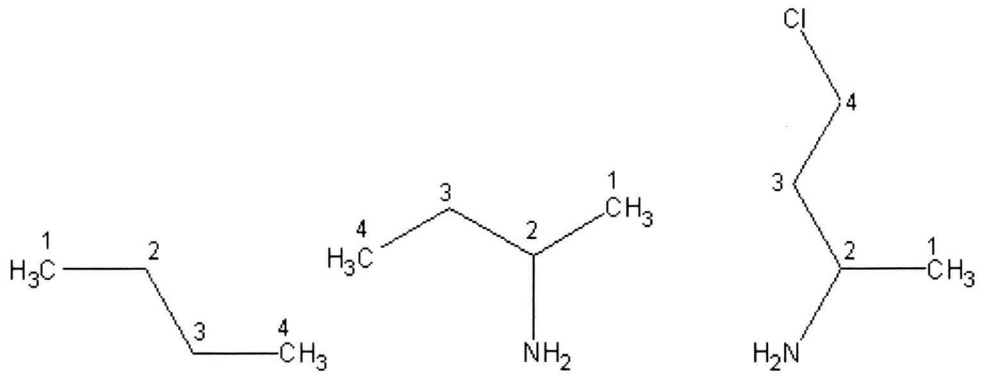
Chemistry has a unique language all to its own. This language is called the IUPAC language, or system. The IUPAC system of language can be quite difficult and confusing to learn, so we will not go into to much depth in this category. What we will discuss is the basic language of chemistry. For starters, you should familiarize yourself with the numbers 1 through 10. These numbers are given in the following table. After you have learned these numbers, practice them using the illustrated structures below.

Mono: 1	Tri: 3	Penta: 5	Hepta: 7	Nona: 9
Di: 2	Tetra: 4	Hexa: 6	Octa: 8	Deca: 10



Dichloromethane Trichloromethane Tetrachloromethane

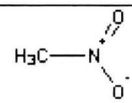
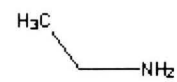
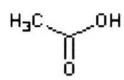
As previously discussed, covalent compounds contain carbon chains, or infrastructures. These carbon chains are numbered so chemists are able to name them. Because the rules that govern the system of numbering can be tricky for beginners to learn, we will not go into to much depth. In the following illustration, butane is shown with correct numbering. Thereafter, another more complicated structure is shown with correct numbering, followed by an even more complicated structure. In each of these examples, the numbering demonstrates how compounds can be numbered and labeled for proper identification.



Butane 2-Aminobutane 2-Amino-4-chlorobutane

Another important tool for being able to name chemical compounds, is knowing the correct functional group. Functional groups are bits and pieces of molecules that have distinctive properties to them. Functional groups play a major role in determining the correct identification for any given compound. Functional groups can be tricky for many beginners to memorize, so we will not go into to much depth here as well. However, we will discuss a few common functional groups that you will encounter in this book. Take a look now at the following table. Notice each unique functional group, and the corresponding chemical compound it is attached to—notice any patterns? The primary functional groups that we will deal with in this book are amine groups.

Some common functional groups

-NO_2 (nitro)		Nitromethane	-NH_2 (amine)		Ethyl amine
-COOH (carboxylic acid)		Acetic acid	-CN (Nitrile)		Acetonitrile
-OH (alcohol)		Ethanol	-CH_3 (Methyl)	CH_4	Methane

As far as the IUPAC system and functional group are concerned, most chemical compounds are identified and named in these manners; although, in some cases, common names have been attributed to many chemical compounds to simply make it easier to identify them. For example, the names of the three chemical formulas illustrated at the top of the page are written in IUPAC nomenclature, but experienced chemists will simply name these compounds methylene chloride (dichloromethane), chloroform (trichloromethane), and carbon tetrachloride (tetrachloromethane). Even though common names are quite common for identifying chemicals, the correct IUPAC name should be given in special cases to correctly identify the compound. For example, 2-amino-4-chlorobutane would not make sense if we simply called it aminochlorobutane. Saying aminochlorobutane does not depict where on the carbon chain the amino functional group is, or the chlorine atom.

7. Conversion factors

SECTION 1: Introduction: A quick lesson in chemistry

For some readers (especially Americans), the metric system (other wise known as the SI system) is vague, or somewhat unfamiliar. 99% of all the units of weight and measurement in this book are given using the SI system; therefore, a translation from one unit to equipment is automatically calibrated in SI units, so even inexperienced persons will not have to worry too much about knowing the SI system. Regardless, try a few conversions of your own just for practice. Example: Convert 150 Celsius into Fahrenheit—Solution: multiply 150 by 1.8 and then add 32. The answer would be 302 Fahrenheit. Example 2: Convert 1.2 gallons into milliliters—Solution: multiply 1.2 by 3,785. The answer would be 4542 milliliters.

To convert	**Into**	**Multiply By**	**To convert**	**Into**	**Multiply By**
Atmospheres	Cm of mercury	76	Liters	Gallons	0.2642
Atmospheres	Mm of mercury	760	Liters	Ounces (fluid)	33.814
Atmospheres	Torrs	760	Meters	Feet	3.281
Atmospheres	In of mercury	29.92	Meters	Inches	39.37
Atmospheres	psi	14.7	Milligrams	Ounces	3.527×10^{-5}
Celsius	Fahrenheit	1.8 + 32	Milligrams	Pounds	2.2046×10^{-6}
Centimeters	Inches	0.3937	Milliliters	Gallons	2.642×10^{-4}
Centimeters	Meters	0.01	Milliliters	Ounces (fluid)	0.0338
Centimeters of mercury	Atmospheres	0.01316	Millimeters	Feet	3.281×10^{-3}
Centimeters of mercury	psi	0.1934	Millimeters	Inches	0.03937
Fahrenheit	Celsius	0.556 – 17.8	Ounces	Grams	28.349527
Feet	Meters	0.3048	Ounces	Kilograms	0.0283
Feet	Millimeters	304.8	Ounces	Milligrams	28,349.5
Gallons	Liters	3.785	Pints (liquid)	Liters	0.4732
Gallons	Milliliters	3,785	Pints (liquid)	Milliliters	473.2
Grams	Ounces	0.03527	Pounds	Grams	453.5924
Inches	Centimeters	2.540	Pounds	Kilograms	0.4536
Inches	Millimeters	25.40	psi	Atmospheres	0.06804
Inches of mercury	Atmospheres	0.03342	Quarts (liquid)	Liters	0.9464
Inches of mercury	psi	0.4912	Quarts (liquid)	Milliliters	946.4
Kilograms	Ounces	35.274	Torr	Mm of mercury	1.0
Kilograms	Pounds	2.205	Torr	Atmospheres	1.316×10^{-3}

SECTION 2: LABORATORY TUTORIAL

Part 2: Laboratory tutorial on techniques and procedures

8. Introduction

Before reading the procedures in this book, you should take a quick lesson in laboratory techniques to better help you understand the procedures discussed in this book. Many of the procedures in this book include distillations, extraction, and recrystallizations, which will all be discussed in detail later on in this section. Un-like A Laboratory History of Chemical Warfare Agents, this book has many exhaustive extractions and recrystallizations to deal with, and you will learn about these in great detail, and will learn to master them (or at least understand them). First of all, laboratory techniques and safety are crucial, especially when carrying out procedures utilizing solvents, and other chemicals. This section will fully teach you about proper laboratory techniques and procedures, and will demonstrate that proper lab techniques will ensure a healthy, and safe working environment. Before we get started, it should be noted that this book is intended to educate people about the chemistry and laboratory techniques involved in the preparation of amphetamines and derivatives. It should be understood that this book represents over 100 years of amphetamine chemistry and development, and is intended for educational purposes only. Students, teachers, researchers, and police officers and other civil authorities may use this book for research and/or training purposes.
Note: Do not attempt to prepare the compounds detailed in this book if you are not of the scientific community.

9. Lab safety

Lab safety is the first step in proper laboratory techniques. For each chemical procedure, read directions carefully, and know precisely what you need to do, before you actually do it. After reading the procedure think about the procedure, and know the hazards associated with it. Know the chemicals used in the procedure and how to properly handle them. Do not attempt to alter the procedure or change chemicals. The best safety is to prevent accidents before they happen.
Carryout all procedures involving volatile chemicals using proper ventilation. Fume hoods work in most cases, but not all. Even in well-ventilated fume hoods, volatile and/or noxious fumes can expand outward contaminating the entire lab. Vapors can travel long distances and cover large areas despite well ventilation.
Under any circumstance, eye protection should be used at all times. Eye protection should include eye goggles that completely seal the eyes; glasses are not proper eye protection. Nitrile gloves, and proper lab coats should be worn at all times.

For general handling of chemicals, including common solvents, reagents, and intermediates, the following checkpoints should be observed:
1) Always remember to wear safety goggles at all times. Clothing and equipment can be replaced, but your eyes can't. Contact lenses or glasses are not a substitute for safety goggles. If you get chemicals in your eyes (liquid, gas, or vapor) immediately flush with large amounts of water.
2) Immediately wash off any chemical you happen to spill on yourself. Most chemicals are dangerous only if they linger, so take action at once. Concentrated sulfuric acid is not very harmful if washed off immediately, and most acids do little or no skin damage if they are immediately washed off with water.
3) In case of an accident such as a fire, save yourself first. Keep fire extinguishers in arms reach, and have an adequate water source within reach. For acid spills, simple baking soda can be used to neutralize it.
4) Avoid open flames in a laboratory setting, and do not smoke in the lab. In the event of a fire, calmly but quickly move away from the burning area. Fight the fire only if you are confident the fire can be extinguished.
5) Do not eat or drink food products while in the lab. Food and drink can become contaminated by accident, and never use laboratory glassware for eating or drinking.
6) Never taste chemicals, and never smell chemicals by sticking your nose right up to the container. Smell chemicals by wafting the vapors with your hand to your nose. Many accidents have occurred when fingers were contaminated in the laboratory and then later used to rub eyes or for eating snacks. Remember to wear gloves at all times. Latex gloves work for

most cases, but in some cases nitrile gloves are recommended. Especially when handling strong acids, or chlorinated solvents. If bare handed, wash hands after touching chemicals and/or their storage bottles.
7) Breathing or handling small amounts of noxious substances does not pose immediate danger, but you should avoid contact with any potentially noxious chemical under all circumstances. Toxic chemicals should be handled with great care, and proper ventilation (fume hoods with maximum settings) should be used. If fume hoods are not available, the toxic chemicals should be handled in well-ventilated rooms with open windows to allow good airflow. Most organic solvents are very volatile and flammable, so proper ventilation should be exercised as well. Always remember, if you can smell a substance, you are breathing it into your lungs.
8) Wear inexpensive clothing when working in a lab. Since there is a possibility of clothing being destroyed in a laboratory accident, a lab coat or an apron should be worn at all times. Do not wear sandals or thong shoes when in the laboratory. Confine long hair and/or loose clothing while in the laboratory. Do not wear shorts, open skirts, blouses, or any other clothing that leaves large areas of skin unprotected.
9) On a final note, never play around with chemicals by mixing or heating them. Always remember, before you mix and/or heat chemicals know what you are doing. Playing around with chemicals can lead to poisonous fumes, fires, and/or explosions.

10. Laboratory equipment

Laboratory equipment is absolutely crucial in the preparation of amphetamines, and cannot be substituted by anything else. Most of the procedures in this book require detailed laboratory glassware, which is very expensive. Laboratory glassware comes in many styles, shapes, and sizes from many suppliers. If you're truly interested in purchasing laboratory glassware, it is best to look into Chinese companies or companies from India; they can provide highly quality glassware for allot less then you would pay in America. Using glassware only requires a few simple rules, which can go as follows:
1) Most laboratory glassware cannot be heated above 500 Celsius. Quartz glass, which is really expensive, is used in procedures where higher temperatures are needed (up to 1200 Celsius) along with the inertness of glass. Steel, nickel, porcelain, or iron crucibles are used for general heating of solids at high temperatures. General laboratory glassware is used for heating liquids because most liquids will never encounter temperatures exceeding 300 Celsius. 2) Never rapidly heat glass to a high temperature. Exposing glassware to high temperatures all at once can cause cracks and breakage. Cooling hot glassware to quickly can also lead to cracks and breaks. Always allow the heated glass to cool to room temperature (by itself) before applying it to cold water baths, ice baths, or dry ice baths. Quartz is an exception. It can be heated to 1000 Celsius and then dipped into water. If you get your hands on any quartz glassware, snatch it up like gold and take good care of it. Quartz glassware can be used instead of ordinary laboratory glassware.
3) The following illustrations show some common laboratory glassware and equipment. Most modern glassware contains ground glass joints. Ground glass joints are outer (male) and inner (female) etched surfaces that stick together forming an airtight seal when pushed together. In some cases sealant grease (commonly called vacuum grease) is applied to the joints to allow for easy disconnection. When connecting adapters, do not push them together to hard. Pushing the joints together to hard may lead to a suction effect between the two adapters. This suction effect can make disconnection of the adapters by hand impossible. In some rare cases the joints can be suctioned together so severely that breakage of the adapters while trying to disconnect them results. If adapters become suctioned together, do not use force to separate them. To separate joints that are suctioned together, simply heat the joint directly with a blue flame from a Bunsen burner, being cautious as not to heat the joint to much—when the joint has been heated for a few quick times, simply pull apart the two adapters using a cloth to protect your hands from the hot glass.

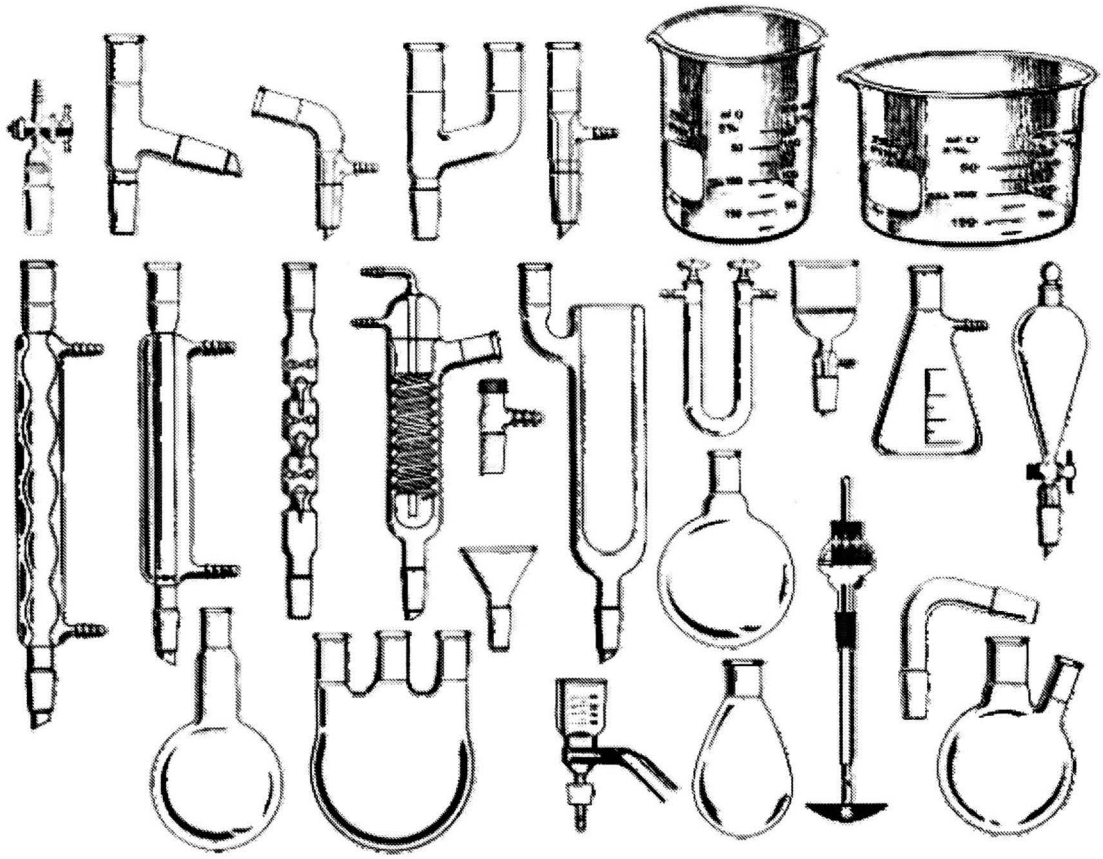


Figure 001. Common laboratory equipment.

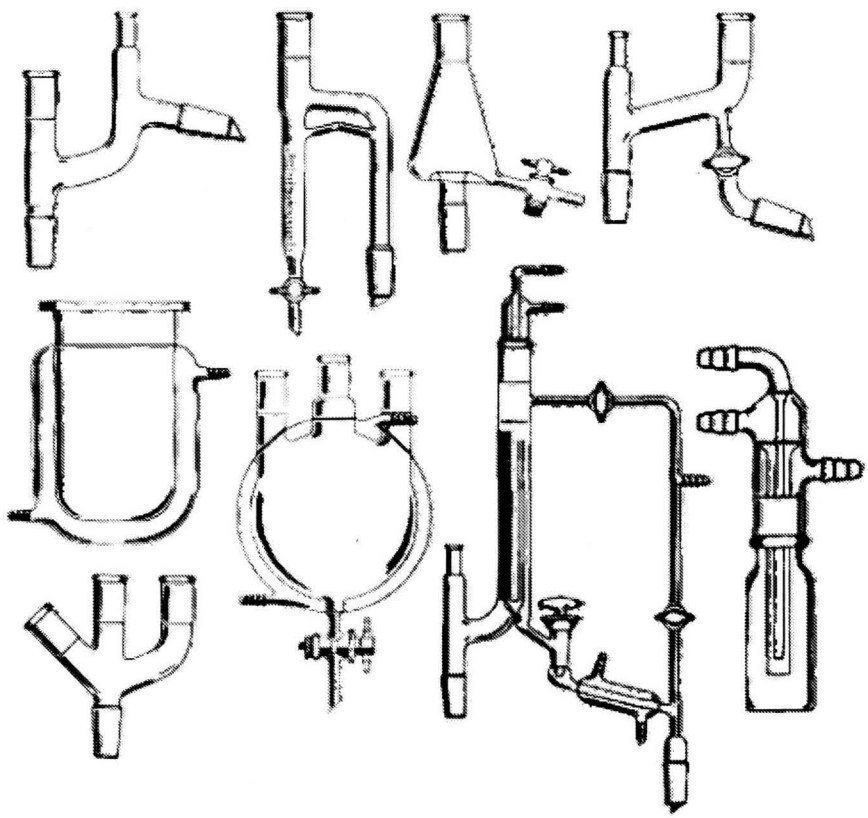


Figure 002. Common laboratory glassware.

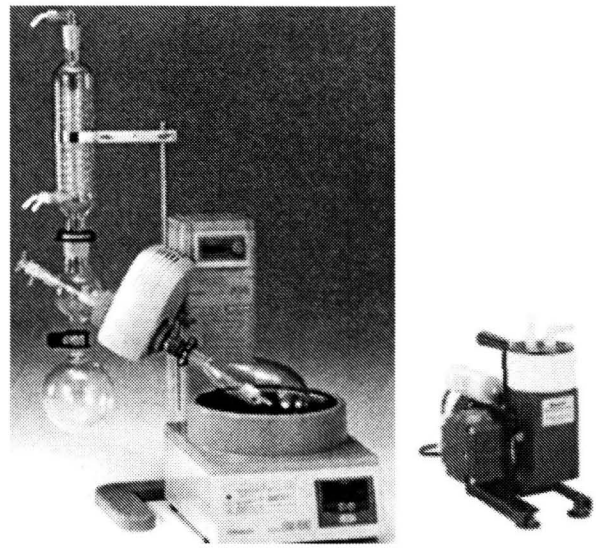


Figure 003. Left: Common laboratory rotary evaporator. Right: Common laboratory vacuum pump.

11. Methods of heating

For heating purposes in the lab, a variety of heating methods can be used. Several factors are involved in determining what method of heating should be used. These factors include the shape and size of the reaction vessel, the desired reaction temperature, and whether the reaction mixture must be stirred at the same time it is heated. The most common methods of heating used in labs are listed below.

1) Free flame

Bunsen burners refer to the term free flame. The Bunsen burner is a commonly used heating device in general chemistry labs, but its use in modern labs is limited. It is very inexpensive to purchase and operate, and permits mixtures to be heated rapidly. Bunsen burners are also commonly used to heat solids. Their use in heating liquids is limited due to potential hazards. Heating liquids with Bunsen burners can lead to violent bumping and foaming. This bumping and foaming can lead to flashovers. In general, never heat flammable liquids with Bunsen burners. When using Bunsen burners, be certain there are no flammable solids, liquids, or vapors in the vicinity. Bunsen burners can be used to heat high boiling liquids such as in the distillation of benzyl chloride, which has a high boiling point—however, never heat volatile chemicals with free flames. Bunsen burners are commonly used in roasting solids and mixtures, such as dehydrating solids as seen when heating Epsom salt to remove its water of hydration, and to form anhydrous magnesium sulfate.

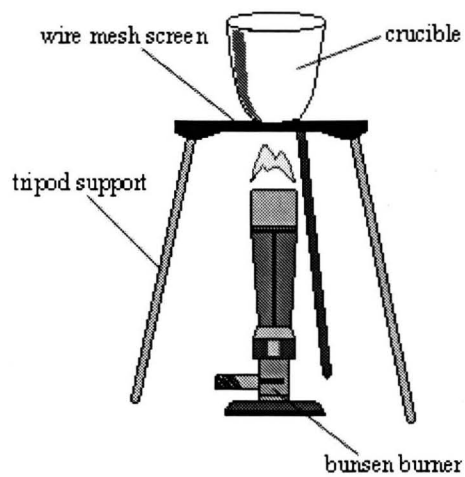


Figure 004. Common laboratory Bunsen burner with support stand.

2) Steam bath

Steam baths are an inexpensive and useful way for heating mixtures up to 100 Celsius. Steam baths can also be used to heat mixtures from 50 to 90 Celsius. Steam baths are very easy to use and operate, and they heat mixtures without blind spots. Blind spots occur when heating is not even. A steam bath is much more useful for heating low-boiling liquids than a free flame, and any vapors which may escape from the distillation apparatus simply dissipate with the steam.

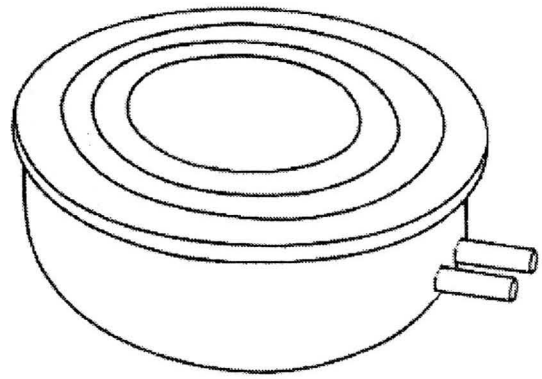


Figure 005. A common steam bath. To use a steam bath, remove enough rings so that a round-bottom flask will rest on a ring enough so to expose it to the steam without falling through.

3) Oil bath

Oil baths are useful for heating mixtures. The contact of the flask with the hot oil heats the flask perfectly because the hot oil completely surrounds the sides of the flask. This results in even heating and effective temperature control. Oil baths are relatively inexpensive and are safe to operate because they lack an open flame. Oil baths are slow to heat, and they cool slowly after use. These are some of the drawbacks associated with oil baths. In addition, the flask retains an oily residue, which is slippery and must be cleaned off.

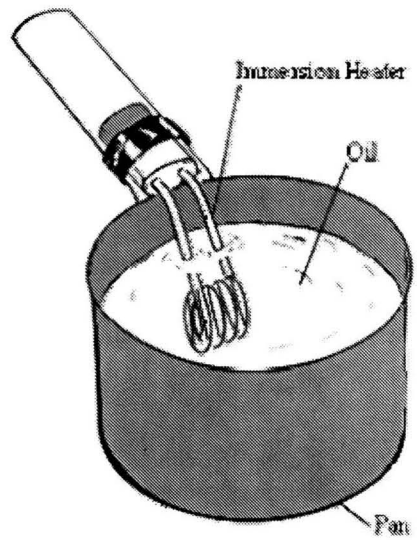


Figure 006. A typical immersion heater used with an oil bath. The flask is immersed about half way into the oil.

4) Electric Heating Mantles

Heating mantles are the most common method of heating round bottom glassware, and they come in a wide variety of shapes and sizes. Sizes ranging from 10 milliliters to a whopping 12 liters are available. The most common sizes are the 250 milliliter,

500 milliliter, and 1000 milliliter models. These models range in price from 80 to 200 dollars. A voltage regulator is usually used to control the heating, and is sold separately. Exercise care in setting the voltage of a heating mantle because too much voltage can lead to undesired temperature. Test the voltage regulator on an empty flask equipped with a thermometer to familiarize you with the temperature settings. Some voltage regulators will clearly indicate the temperature. A label is usually attached to the heating mantle, which indicates the maximum safe voltage. Note: A heating mantle designed to tolerate a maximum of 20 volts quickly burns out if 120 volts is applied. Read the maximum tolerances aloud for your heating mantle before using it.
Most 100 to 500 milliliter heating mantles tolerate a full 120-volt input, and some large mantles even require two voltage regulators. On a final note, be certain the heating mantles size is appropriate for the flask being used.

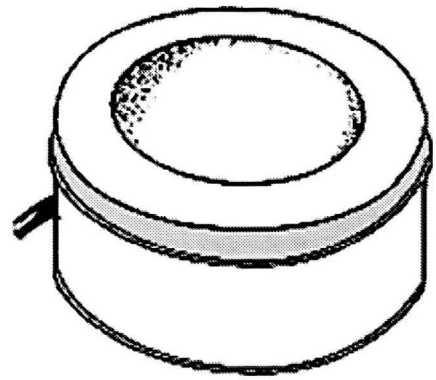


Figure 007. A classic heating mantle.

5) Hot Plates

Hot plates are by far the most common method of heating flat bottom laboratory glassware. Hotplates are exclusively used in heating Erlenmeyer flasks and beakers. Many hot plates come doubled with a magnetic stirrer and are usually called hot plate/stirrers. These hot plate/stirrers are very useful in the heating and the simultaneous mixing of liquids. Some hot plates come without magnetic stirrers. Laboratory hotplates heat relatively slow and they cool slowly, but their energy efficient and they maintain the desired temperatures for indefinite time.

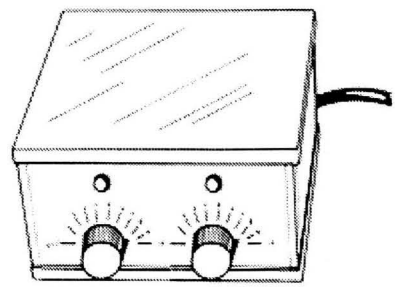


Figure 008. A common hot plate with a magnetic stirrer. Most hot plates double as magnetic stirrers.

12. Methods of Cooling

Cooling is often required during a chemical reaction in order to maintain proper reaction temperatures. Not properly cooling reaction mixtures can lead to conditions including evolution of poisonous gases, decomposition of products, and unwanted side reactions. Cooling baths are cheap and readily available. Dry ice is readily available and is used to make excellent cooling baths.
Cooling is not as easy as it may appear. In some ice baths the ice will melt rapidly during the chemical reaction. Ice that rapidly melts must be continuously refilled in order to maintain proper reaction temperature.
Cooling baths should be at least three times the volume of the reaction flask. For example, if using a 1-liter flask to contain the reaction mixture, a 3-liter container should be used to house the 1-liter flask. Before adding the cooling agent (ice water, ice, or dry ice) to the bath, make sure the 1-liter flask is seated in the bath container. Then fill the container with the cooling agent. The 1-liter flask should be submerged as far as possible into the ice bath. In other words, 80% of the total height of the 1-liter flask should be submerged in the cooling bath. In some cases the flask being cooled will displace the cooling agent (cold water,

or ice water) causing it to float and possible tip over. Lead rings, which are cheap and commercially available, make useful weights to keep the reaction flask seated in the cooling bath.

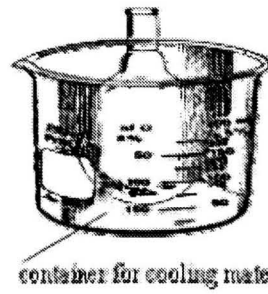


Figure 009. Setup for cooling bath. The container can be used to house any cooling medium.

1) Cold water bath

Simple cooling utilizes a cold-water bath. Cold-water baths are used to keep the reaction temperature from 15 to 50 Celsius. In some cases the water bath will have to be quickly drained, and then refilled with cold water in order to maintain the desired reaction temperature. In most cases cold-water baths are used for general long-term temperature control.

2) Ice water bath

Ice water baths are commonly used to keep reaction temperatures around 5 to 30 Celsius. Ice water baths are used in place of cold-water baths where long term cooling, but a slight colder temperature is needed.

3) Standard ice bath

The standard ice bath is the most common method of cooling reaction mixtures. This method of cooling can produce temperatures of 0 to 20 Celsius. Ice baths are composed of chopped up pieces of ice, and the ice should be finely crushed so that it adheres to the wall of the reaction flask as much as possible. Remember to place the reaction flask into the empty bath container before adding the ice. As the cooling proceeds the ice may melt rapidly, moderately, or slow. If the ice is melted, drain off the water and then add more finely crushed ice. Continue the process as many times as needed. Depending on the time and conditions, the ice may not have to be replaced.

4) Salt/ice bath

The salt/ice bath is a modified version of the ice bath. Depending on the type of salt used, salt/ice baths are very useful for producing temperatures ranging from –55 to 0 Celsius. To prepare a salt/ice bath, simply mix the finely crushed ice with 20% of its weight in salt. Salt/ice baths can maintain their temperatures for varying amounts of time depending on the heat evolved during a particular chemical reaction, time, and/or other conditions. In some procedures the salt/ice bath will have to be replaced with a fresh batch. When the salt used is potassium chloride the temperature achieved will be around –10 to 0 Celsius. When the salt used is sodium chloride the temperature achieved will be –20 to 0 Celsius. When the salt used is anhydrous magnesium chloride the temperature achieved will be –30 to 0 Celsius, and when the salt used is calcium chloride hexahydrate the temperature achieved will be –55 to 0 Celsius.

5) Dry ice/acetone bath

Dry ice baths are very common in the modern laboratory. Dry ice is readily available and can achieve temperatures of –70 to –30 Celsius. Dry ice is seldom used along for cooling purposes due to its volatility. It is usually used in combination with a solvent. The solvent is normally acetone, but ethanol, ethyl acetate, or ether can be used. To use a dry ice/acetone bath, add the dry ice to its same weight in acetone (50/50) and then place this mixture into the bath container. Then place this dry ice/acetone filled bath container into a second yet larger container and then fill this second larger container with ice/salt. The second container bath acts like an insulator to the inner bath container giving longer life to the dry ice/acetone bath. The dry ice bath may rapidly deplete if you withhold the second cooling bath. For short-term cooling and use, the second cooling bath will not be needed. For long term cooling, withhold the second cooling bath and place the dry ice/acetone bath into a refrigerator freezer.

6) Cooling tricks of the trade

One method of cooling is to place the reaction apparatus, flask, or beaker into a refrigerator or freezer (as long as it fits). This allows for complete cooling without refilling containers with ice or cold water. A major draw back to doing this is a lack of ventilation. In some procedures highly poisonous and corrosive gases are evolved and hence must be properly vented. If a procedure is relatively free from toxic or corrosive emitions, the apparatus can be placed into a freezer or refrigerator if it fits. Refrigerators and freezers are also very handy when having to store reaction mixtures for several hours or several days. Simply place the reaction flask into the refrigerator or freezer and then cool for the amount of time needed. This eliminates the need for ice baths and the like.

13. Extraction

Extraction is a major part of many chemical procedures, and is usually conducted before the recrystallization process. Extraction is used to "separate" a product from a reaction mixture. The reaction mixture is merely shaken with a certain solvent multiple times. During this shaking, the desired product in the reaction mixture is dissolved into the solvent. The solvent is then removed from the reaction mixture, and the product recrystallized from the solvent.
The volume of solvent used is dependent on the desired products solubility in it. When the volume of the solvent has been determined, it is broken into small portions, and then each portion is shaken with the reaction mixture independently. After all the portions have been shaken with the reaction mixture, they are combined and then the product is recrystallized. For the chemical procedures in this manual, the solvent, quantity, and volume size of each portion is given in detail.

1) Funnel Size

The size of the seperatory funnel is of practical consideration when carrying out the extraction process. A seperatory funnel is the piece of glass traditionally used in extraction. In order to leave room for shaking the solution the funnel should be 30 to 50% larger than the total combined volume of liquid. For example, use a 250-milliliter seperatory funnel when extracting 100 milliliters of reaction mixture with 50 milliliters of solvent. If you are extracting large volumes of liquid, and you don't have a proper sized seperatory funnel, simply divide the reaction mixture into smaller portions and do the same for the solvent portions.

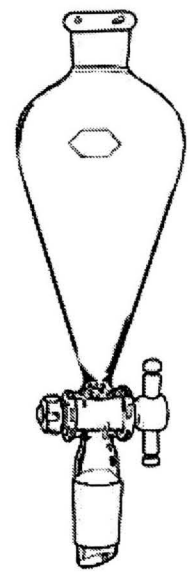


Figure 010. A standard laboratory seperatory funnel.

2) Performing the Extraction

The first step in extraction is to pour the reaction mixture and the solvent into the seperatory funnel. A two-layer mixture will result. Which layer is what depends on the densities of the chemicals in the reaction mixture verses the density of the solvent. If the density of the solvent is greater then the chemicals in the reaction mixture, the solvent will be the bottom layer. If the opposite is true, the solvent will be the upper layer. For example, when a water solution is to be extracted with two portions of methylene chloride, the water solution and the first portion of methylene chloride are placed into the seperatory funnel (make sure the stopcock is closed). A two-layer mixture results. The methylene chloride will be the bottom layer because methylene chloride is denser then water. Then shake the mixture for several minutes. Afterwards, drain-off the bottom methylene chloride

layer only, leaving the water solution in the seperatory funnel. After the bottom methylene chloride layer is removed, pour the second methylene chloride portion into the seperatory funnel and then begin shaking. Then once again, drain-off the bottom methylene chloride layer. At this point the water solution has been successfully extracted. Both drained-off methylene chloride portions can then be combined (if not already done so), and the product recrystallized. Note: If sulfuric acid is present in the reaction mixture, the methylene chloride will always be the upper layer. Sulfuric acid is denser then methylene chloride. Which layer is what will be described for each extraction process in this book. Certain solvent combinations (a water solution of sodium hydroxide and chloroform) lead to emulsions when shaken together. Emulsification cannot always be anticipated, so choose the solvent wisely, or wait along time after shaking for the emulsion to dissipate.

1. Place the reaction mixture to be extracted into a seperatory funnel (make sure the bottom stopcock is closed).
2. Add the solvent portion slowly to the seperatory funnel.
3. Stopper the seperatory funnel, and then begin shaking the funnel for a few minutes.
4. After shaking for a few minutes, allow the two layers to completely settle, and then properly vent the funnel as shown in the following illustration. Then slightly open the bottom stopcock and slowly drain-off the bottom layer. If the upper layer is the solvent, the bottom reaction mixture layer will have to be drained off first, and then poured back into the same seperatory funnel after the upper solvent layer has been drained off. If the bottom layer is the solvent, simply drain it off only, and leave the upper reaction mixture layer.
5. After the appropriate layer or layers have been drained off, and the reaction mixture is the only liquid in the seperatory funnel, add the second portion of the solvent and repeat steps 1 through 5.
6. Repeat steps 1 through 5 as many times indicated in the procedure. For example, if an extraction calls for three portions of methylene chloride, conduct steps 1 through 5 three times.
7. After the number of extractions has been completed, combine all drained-off solvent portions (if not already done so).

Note: In some cases the reaction mixture will be very dark in appearance, and when extracted, forms another dark appearance with the solvent making the phase boundary between upper and bottom layers hard to see. If this happens, hold the seperatory funnel up to a light, or use a flashlight.

Note: While shaking the funnel, vapors from the reaction mixture and/or solvent can increase pressure inside the seperatory funnel. Proper venting of the seperatory funnel is necessary in order to relive this pressure. To properly vent a seperatory funnel, rest the funnel in one hand while grasping the glass stopper. Then tilt the funnel so that the stopcock end is pointed up and away from anyone including yourself. After which rotate the stopcock to the open position. Be certain that the level of the liquid is below the stopcock opening so that none is forced out when the stopcock is opened.

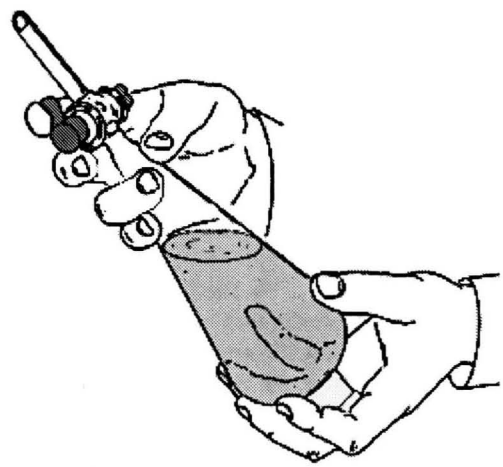


Figure 011. Correct way of venting a seperatory funnel.

3) Draining the funnel

After shaking the funnel, the layer or layers must be drained off. To do this, simply place the seperatory funnel into a ring stand supported by a base support. The stopper must be off in order to drain the funnel, and before opening the stopcock remove the stopper. Attempting to drain the funnel before removing the stopper can result in a vacuum making it difficult to remove the stopper.
When draining the bottom layer, the speed should be adequate as to not over drain. Over draining means to accidentally drain-off some the upper layer. The opening of the stopcock (either fully or partially open) is determined as the phase boundary of the upper liquid approaches the stopcock. When the phase boundary is far away, draining can be done rapidly. When the phase boundary approaches the stopcock, the drain speed should be reduced to a drip.

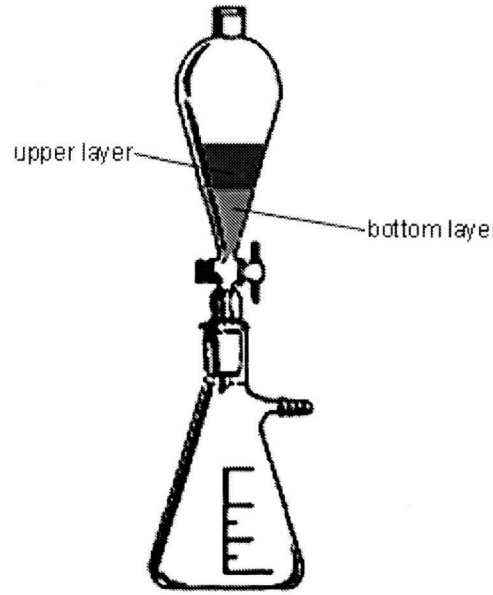


Figure 012. Seperatory funnel positioned for draining.

14. Salting Out

In some cases, an organic compound (usually a liquid) dissolved in water can be precipitated by the addition of sodium chloride, sodium sulfate, or magnesium sulfate. These salts have a much higher affinity for water then most organic compounds, so they tend to dissolve in the water leaving the dissolved organic compound with no room to remain dissolved. The lack of space causes the organic compound to precipitate (organic liquids form a second layer). Water solutions of isopropyl alcohol (rubbing alcohol) for example, can be salted out by the addition of sodium chloride to the mixture followed by rapid shaking of the mixture. The quantity of sodium chloride used is determined by the alcohol concentration. The weaker the concentration is, the more salt is needed. After shaking, a two-layer mixture results. The isopropyl alcohol will be the top layer, and the brine solution the bottom. Try it out for yourself, i.e., salt out a sample of rubbing alcohol using a seperatory funnel and salt.

15. Recrystallization, product recovery, and filtration

Recrystallization is a very important tool for purifying solids. In recrystallization, solubility differences allow solids to be separated from each other and recovered from the solvent. In the recrystallization process, molecules slowly deposit from solution and attach to each other to form crystals. As the aggregates of crystals grow large enough, they precipitate. After precipitation, the solids can be recovered by filtering them off.
Choosing the appropriate solvent is the most crucial aspect of the recrystallization process. The best solvent for recrystallization is one in which the material is less soluble at room temperature but more soluble when hot. At higher temperatures, solvents that form super saturated solutions with certain solids meet this requirement.
Solvent choice is also governed by another important factor, the ease of solvent removal. Solvents with low boiling points are preferred because their removal is easy. A third consideration in selecting a solvent is the temperature at which the solvent solidifies. Benzene was once widely used in recrystallization, but when placed in an ice bath, crystals of benzene would also precipitate (benzene crystallizes at 6 Celsius). A final consideration in choosing a solvent is reactivity. Obviously a solvent that reacts with a solid cannot be used for recrystallization.
Recrystallization depends on super saturation. Super saturated solutions are formed when mixtures containing the dissolved solid and the solvent (or solvent mixture) are heated, or evaporated. When the mixture is heated, the solvent begins to evaporate, as the evaporation proceeds, the concentration of the dissolved solid(s) begins to increase. During this evaporation, the solids become *over dissolved* leading to the super saturated solution. When tiny crystals start forming on the surface of the mixture during heating, super saturation has been reached. When the supersaturated solution is cooled, recrystallization begins and some of the dissolved solid precipitates as crystals. Not all the solid will precipitate out on cooling. After the supersaturated solution has cooled for some time, equilibrium sets in restoring the original solubility of the mixture. The only difference is that some of the solvent and solid have been removed. The precipitated crystals are then collected by filtering the mixture. To recover more solid, the mixture must be re-heated and allowed to evaporate to the point of super saturation again.

1) The recrystallization process

The recrystallization process is simple. Boil the mixture that contains the dissolved product and the solvent (or solvent mixture). The mixture can be placed into a distillation apparatus and distilled at the boiling point of the solvent to collect the solvent. Using a distillation apparatus is preferred rather then just boiling-off the solvent, which would be a waste of solvent. Boil off the solvent until super saturation is achieved. When tiny crystals begin to form on the surface of the mixture, super saturation has been achieved. Then remove the heat source (turn off the heat source) and allow the mixture to cool to room temperature. Afterwards, place the mixture into a cold-water bath or ice bath for thirty minutes. After which, remove the cooling bath, and then filter-off any precipitated product. Then place the filtered mixture back into the same distillation apparatus, and re-distill again until a super saturated solution is achieved. When super saturation is achieved, remove the heat source, and allow the mixture to cool to room temperature. Afterwards, place the mixture into a cold-water bath or ice bath for thirty minutes. Then remove the cooling bath, and then filter-off any precipitated product. Then place the filtered mixture back into the same distillation apparatus, and distill until a super saturated solution is achieved. When it is achieved, remove the heat source, and allow the mixture to cool to room temperature. After which, place the mixture into a cold-water bath or ice bath for thirty minutes. Then filter-off any precipitated product. At this point much of the solvent has been removed by distillation, and much of the product has been recovered. The remaining mixture is called the mother liquor, and can be recycled to a future recrystallization of the same product (using the same solvent or solvent mixture). This process of boiling, cooling, and filtering should be repeated as many times as necessary. When recrystallizing a product from a solvent or solvent mixture, continue the process until 90% of the solvent has been removed. Depending on the solubility of the product, continue the recrystallization process until 75 to 98% of the solvent has been removed. After most of the product has been collected, it can then be washed. To wash a solid product, simply leave it in the filtering funnel, and then pass an inert solvent over it many times. Choose a solvent that does not dissolve the product. Water is usually used to wash organic solids.

2) Seed Crystals

In some cases recrystallization of super saturated solutions can be initiated with a seed crystal. A seed crystal is simply a small crystal of the product. It is added to the super saturated solution, and the dissolved product begins to grow on the seed crystal. The seed crystal induces recrystallization by giving the dissolved product a surface from which to grow on. The recrystallization of the product stops when equilibrium of the solution is reached.

3) Recovering the product through low heat or no heat evaporation

In most modern labs, the recrystallization process is passed over by a rotary evaporator. A rotary evaporator, as pictured earlier in this section, is the most common method of recovering dissolved product. To use, the reaction mixture is placed there into, and then a vacuum is applied. The flask containing the reaction mixture is partly submerged in a water bath, and the necessary amount of heat is applied. Because liquids have decreased boiling points with decreasing pressure, solvents can be removed at much lower temperatures thanks to the vacuum. This process is similar to vacuum distillation. The great thing about rotary evaporators is their ability to run for hours on end without having to interact, monitor, or take part in the process. Simply insert the reaction mixture, apply the necessary heat, attach the vacuum, and let the machine do the rest of the work. Rotary evaporators sell for about $3,000 to $10,000 a piece.

The oldest method of product recovery is placing the reaction mixture into a crystallizing dish, or shallow pan, and then allowing the solvent(s) to air evaporate. This method is a good idea for crystallizing stable, light insensitive products, where good crystal size is desired; for example, allowing a solution containing sodium chlorate to air-evaporate, large brilliant crystals of the chlorate are obtained. If this same solution was recrystallized, or evaporated under vacuum, usually small crystals of the chlorate are obtained. The problem with air-evaporation is the amount of time required, especially if the product is hygroscopic. In some examples, air-evaporation is impossible. Examples include zinc chloride, lithium perchlorate, and calcium chloride. These substances are so hygroscopic that placing the dry crystals into a beaker will produce a self-induced aqueous solution on standing after several days or weeks due to moisture absorption from the air. In warm dry climates, such as dessert climates, air-evaporation has it advantages. Good crystal size can be rapidly achieved by allowing reaction mixtures to air-evaporate in the sunlight.

16. Filtration

Filtration and recrystallization run hand in hand. When a product precipitates, it must be collected. Filtration is the most common method of collecting precipitated products. The two methods of filtration include gravity, and vacuum. Vacuum filtration is the most common method of filtering in the lab, and it is also the fastest.

1) Gravity filtration

Gravity filtration is the oldest and slowest method of filtering. In most regards gravity filtration should be avoided due to the slow nature. In many examples gravity filtration can take hours, and even days. Even so, gravity filtration is useful for removing charcoal, which is difficult to remove from mixtures when using vacuum filtration.

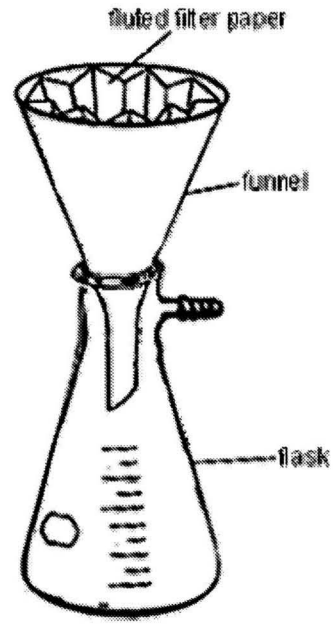


Figure 013. Apparatus for gravity filtration.

Gravity filtration is sometimes used to remove impurities rather then to collect a precipitated product. In this case the filtration takes place with the use of a filter aid. A filter aid is an insoluble substance used to absorb impurities. Some examples of filter aids include Celite, silicon dioxide, sand, zeolites, and even pebbles. Celite is a diatomaceous earth material that is most commonly used. Although filter aids can be used to speed up the filtration of finely divided precipitates, which otherwise get stuck in the tiny holes of the filter paper.

2) Fluting Filter Paper

Laboratory filter paper used in modern labs is usually circular in nature, so fluting the paper is necessary. Fluted filter paper is superior to flat filter paper because fluted filter paper allows for better airflow between the funnel wall and the fluted filter paper.

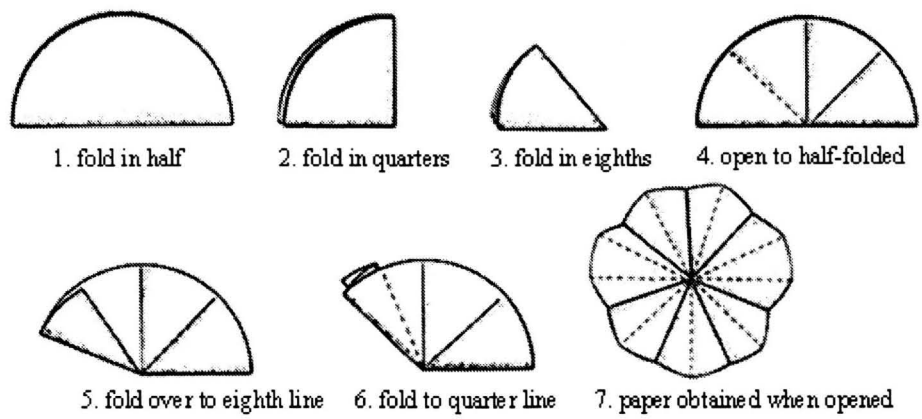


Figure 014. Fluting filter paper.

3) Vacuum Filtration

Vacuum filtration is definitely the method of choice for filtration, and it is the most common method. Vacuum filtration is superior in that suction is used to force the liquid through the filter paper allowing for rapid filtration. Precipitates can be

recovered quickly and easily. After the precipitate has been recovered, it can then be vacuum dried. Vacuum drying is simply allowing suction to continue after the liquid has been removed. The suction creates an excellent airflow, which dries the collected precipitate as it flows. Ten to twenty minutes is adequate time to dry any product.
When first starting the filtration process, the vacuum will suck some of the product into the flask. The contents of the flask should then be re-filtered to ensure no product loss. The suction force is generated by a vacuum pump, which is commercially available in many styles and sizes; hand driven pumps can be used as well. Note: The suction force should not be too great. Placing your hand completely over the funnel until the suction grips your hand moderately indicates the proper suction. Never underestimate the power of a vacuum.
A Buchner funnel is used in vacuum filtration, and is a glass or plastic funnel. Plastic Buchner funnels are composed of two pieces. The funnel cup makes up the top piece, and the stem makes up the bottom piece. Glass Buchner funnels are composed of one or two pieces, and some come with glass joints. To use the funnel simply attach it to the filtering flask (see illustration below), and then place a piece of round filter paper into the bottom of the funnel. The filter paper is simply held in place by gravity and the suction force. Before filtration begins, lightly moisten the filter paper with water or fresh solvent (the solvent used should be the same as in the mixture being filtered, or an inert solvent that does not dissolve the precipitate). Once the precipitate has been filtered and dried, simply remove the suction source and then casually remove the filter paper from the Buchner funnel. Then gently scrape off the product from the filter paper.

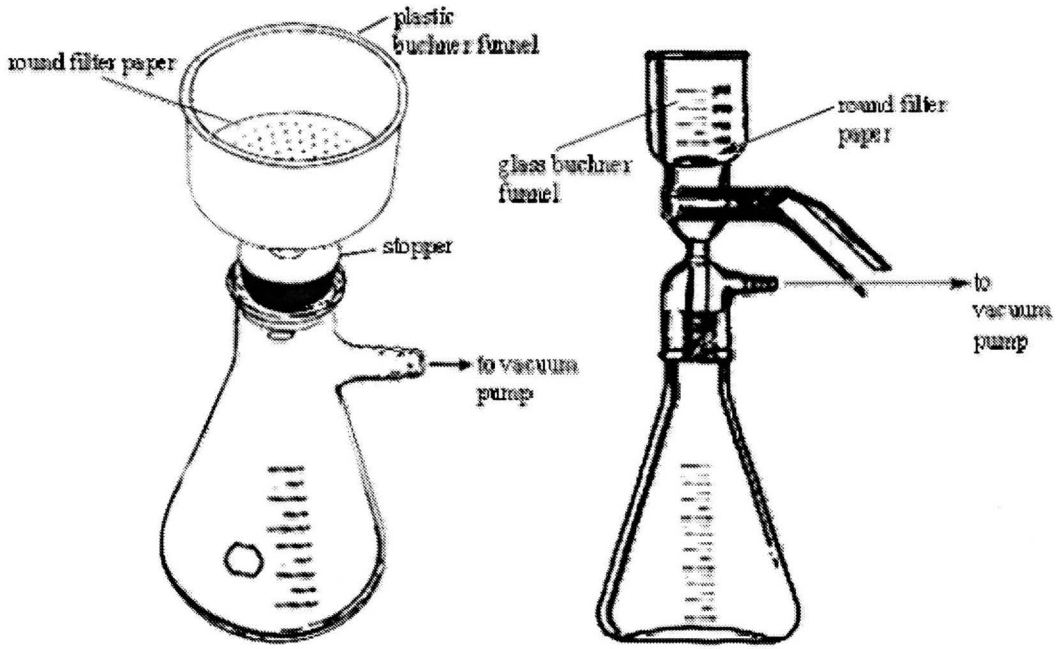


Figure 015. Left: Plastic Buchner funnel. Right: All glass set-up.

17. Washing liquids and solids

Solids are easily washed by passing water, or the desired solvent over the solid product, which is contained in the filter funnel. For washing solids in this way, vacuum filtration should be used. Washing solids in this way using gravity filtration is a long and time consuming process. Obviously, do not wash the filtered-off solid with any liquid that reacts with, or dissolves the solid product.

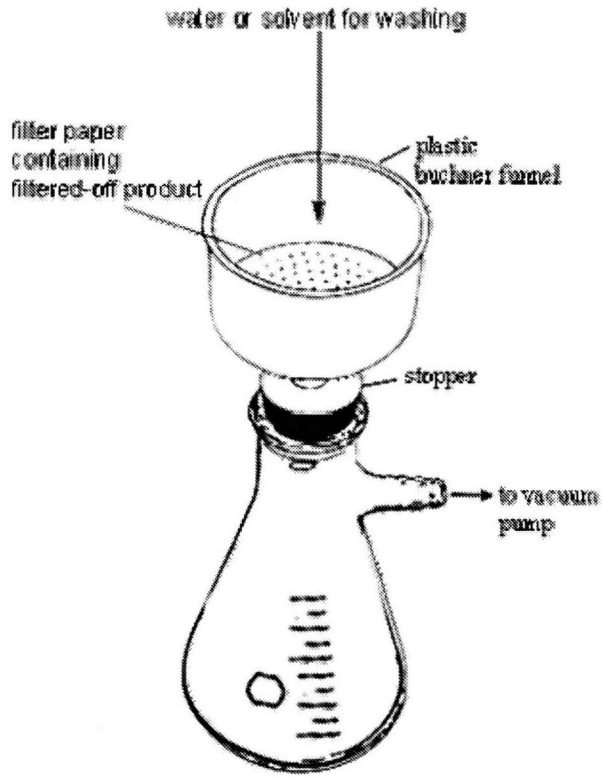


Figure 016. Washing a solid product.

Another method of washing a solid product is to place it into a beaker, and then add an excess of water or solvent, stirring the mixture for several minutes, and then allowing the mixture to stand long enough for the solid product to settle. After the solid product settles, much of the water above the settled solid product can be removed by carefully tilting the beaker, and pouring it off. This method of washing is useful for washing large amounts of water-insoluble product.
Washing liquids is done in a similar manner as just described. For washing a liquid, usually with water, place the liquid into a beaker, and then add the desired amount of water. Thereafter, stir the mixture for several minutes, and then allow the mixture to stand. After the two-phase mixture has settled, remove the water layer either by using a seperatory funnel, or by pouring it off.

18. Drying agents and drying liquids

Water is called the universal solvent, but in many cases its considered to be an impurity. After the extraction process, the combined solvent portions sometimes contain a small amount of water. This water is removed by treating the combined solvent portions with an inert drying agent. The drying agent simply absorbs the water. The most commonly used drying agents are listed below.

1) Anhydrous sodium sulfate

Anhydrous sodium sulfate is the most common general-purpose drying agent. It is inexpensive and has a very large capacity of absorption because it can form a decahydrate. Anhydrous sodium sulfate is relatively inert, and it does not react with most organic compounds. Anhydrous sodium sulfate can be regenerated from used sodium sulfate by heating to 200 Celsius for 1 hour.

2) Anhydrous magnesium sulfate

Anhydrous magnesium sulfate is the second most commonly used drying agent. Similar to anhydrous sodium sulfate, it to has a high capacity for absorption, and low cost. Although unlike anhydrous sodium sulfate, it has a faster drying rate, but is more reactive. It can be regenerated in the same manner as anhydrous sodium sulfate.

3) Calcium chloride

Calcium chloride is very inexpensive, and is an excellent drying agent. Its very high capacity and rapid drying ability makes it the reagent of choice for drying hydrocarbons, chlorinated solvents, halogens, and ethers. Unfortunately, calcium chloride is much more reactive than either sodium or magnesium sulfate and thus cannot be used to dry amines, alcohols, acid gases, or ammonia. It can be regenerated in a similar manner as anhydrous sodium sulfate.

19. Distillation

Distillation is a very common method for purifying liquids. Atmospheric distillation (general distillation), vacuum distillation, and steam distillation are the three common methods of distillation. Atmospheric distillation takes place at atmospheric pressure, which means the distillation apparatus is open to the air. Vacuum distillation utilizes reduced pressure to distill a liquid at lower temperature. Vacuum distillation is commonly used to distill liquids, which tend to decompose at their atmospheric boiling points. Vacuum distillation is also used to conveniently distill liquids with relatively high boiling points at a much more efficient temperature. Steam distillation is similar to atmospheric distillation, but steam is used to promote volatility. Steam distillation only works on liquids or solids which are volatile with steam.

1) Atmospheric Distillation (general distillation)

Atmospheric distillation is the most common of the three methods of distillation. The following illustration shows a common distillation apparatus. When liquids are heated they become volatile. The degree of volatility depends on the amount of heat applied to the liquid, the pressure, and the chemicals boiling point. When enough heat is applied to the liquid, the liquid begins to boil. When a liquid boils, intermolecular forces within the liquid break, and the molecules there after convert into the gas phase. During the distillation, this gas passes over into a condenser, where it is condensed back into a liquid by applying a cooler temperature to the gas. A condenser usually filled with circulating cold water acts as the cooling force. When the gas is cooled, it reforms back into a liquid, and then gravity pulls it into a receiver flask where it collects. A typical distillation produces 1 to 50 milliliters of liquid per minute. Most distillations take hours, depending upon the volume of liquid being distilled, and the concentration. Concentrated solutions distill much faster then dilute ones.

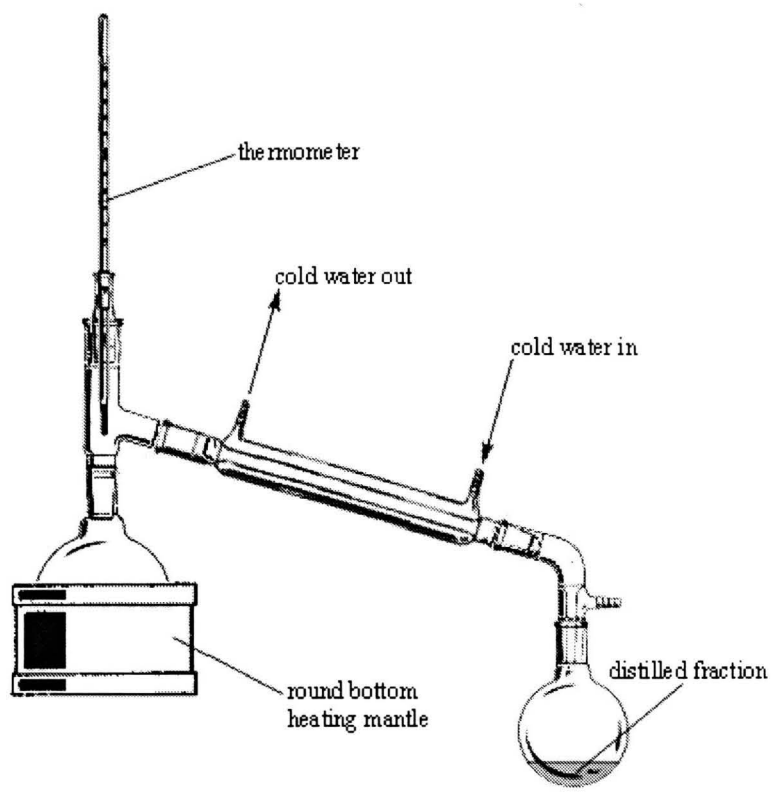


Figure 017. Standard atmospheric distillation apparatus—can be used for most distillation processes.

2) Vacuum Distillation

Vacuum distillation is another common method of distillation, and is very useful in the purification of high boiling compounds. Many compounds have boiling points that make purification through ordinary distillation difficult. As well, some compounds

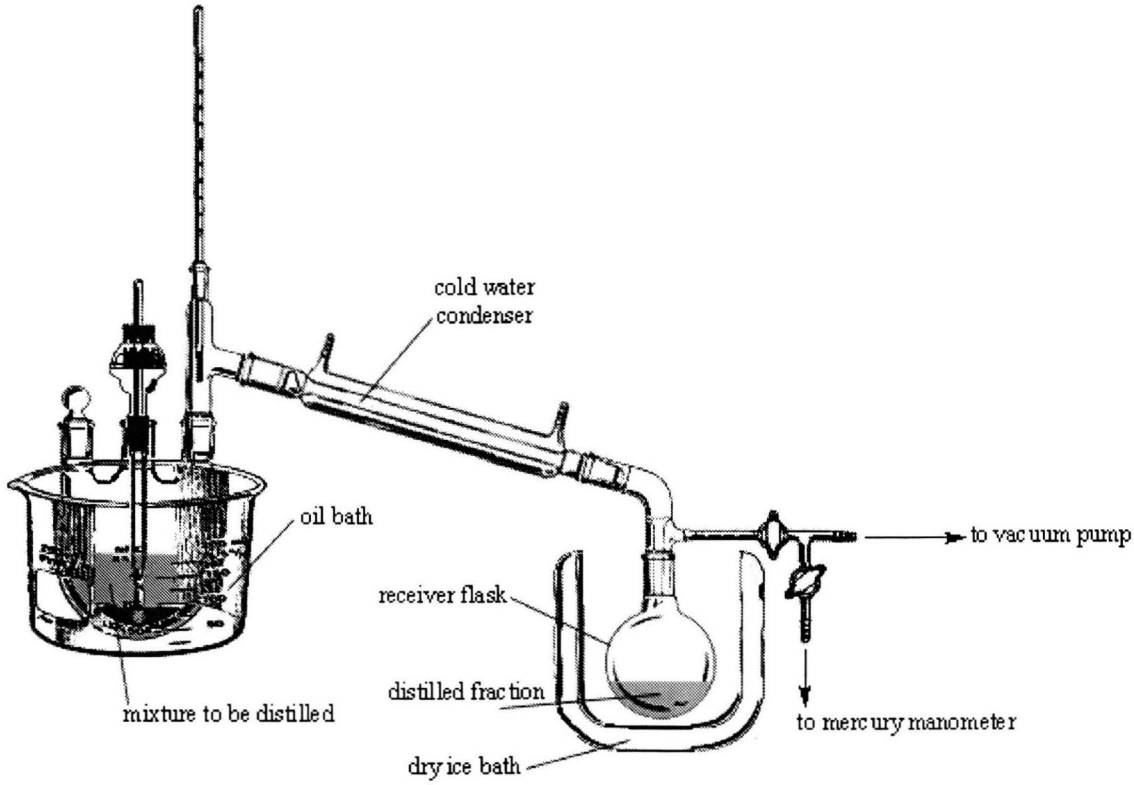


Figure 019. Common vacuum distillation apparatus with electric stirrer. A mercury monometer is connected to the system to monitor the vacuum. A common mercury manometer can read vacuums down to 1 millimeter of mercury or less. Note: The oil bath is pictured without the immersion heater.

3) Steam Distillation

Steam distillation is the third most common method of distillation. The following illustration shows a common steam distillation apparatus. Steam distillation takes advantage of the volatility force of water upon certain solids or liquids. Many liquids and solids are volatile with steam, which means they partially volatize when contacted with steam without actually converting to gas. The steam merely acts as a carrier picking up the solids or liquids, and then carrying them over in a conventional distillation manner. The products being steam distilled, whether soluble in water or not, are collected in the receiver flask.

Steam distillation works by the interference of hot gases upon solids or liquids. The solids partially volatize forming finely divided micro particles, and liquids partially volatize forming micro sized droplets of liquid (a mist). The gaseous water then takes-up these finely divided micro particles or small droplets of liquid, and carries them over in a conventional distillation manner. Think of smoke for example, it appears to be a smooth flowing gas when actually it's a mixture of finely divided micro particles mixed in with colorless gases. Most steam distillation apparatuses generate their steam internally, but some use steam provided from a steam line.

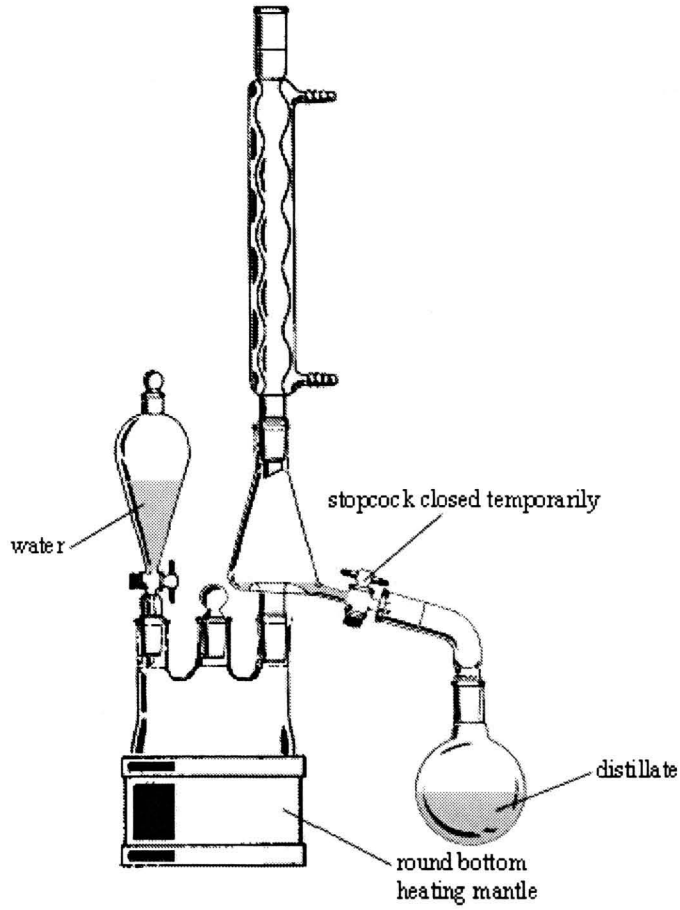


Figure 020. Common steam distillation apparatus with steam generated internally.

20. Apparatus design and function

Apparatus design is crucial in properly carrying out chemical reactions. Without proper glassware and corresponding apparatus, most chemical reactions would be difficult to carryout, control, and monitor. Other factors which make apparatus design important are, controlling and containing the release of flammable or noxious fumes, temperature control, and maintaining proper reaction conditions. The following apparatus designs are not set in stone, and can vary widely; however, all apparatus design has a similar foundation to it, meaning adapters, thermometers, tubes, ect., ect, should be similar, i.e., should be utilized in the same manner where applicable. For example, if bubbling a gas into a reaction mixture, the tube being employed for the addition should be submerged into the reaction mixture, but exactly how long the tube is, or exactly what joint of stopper it is held to can vary.

1) Reaction apparatus

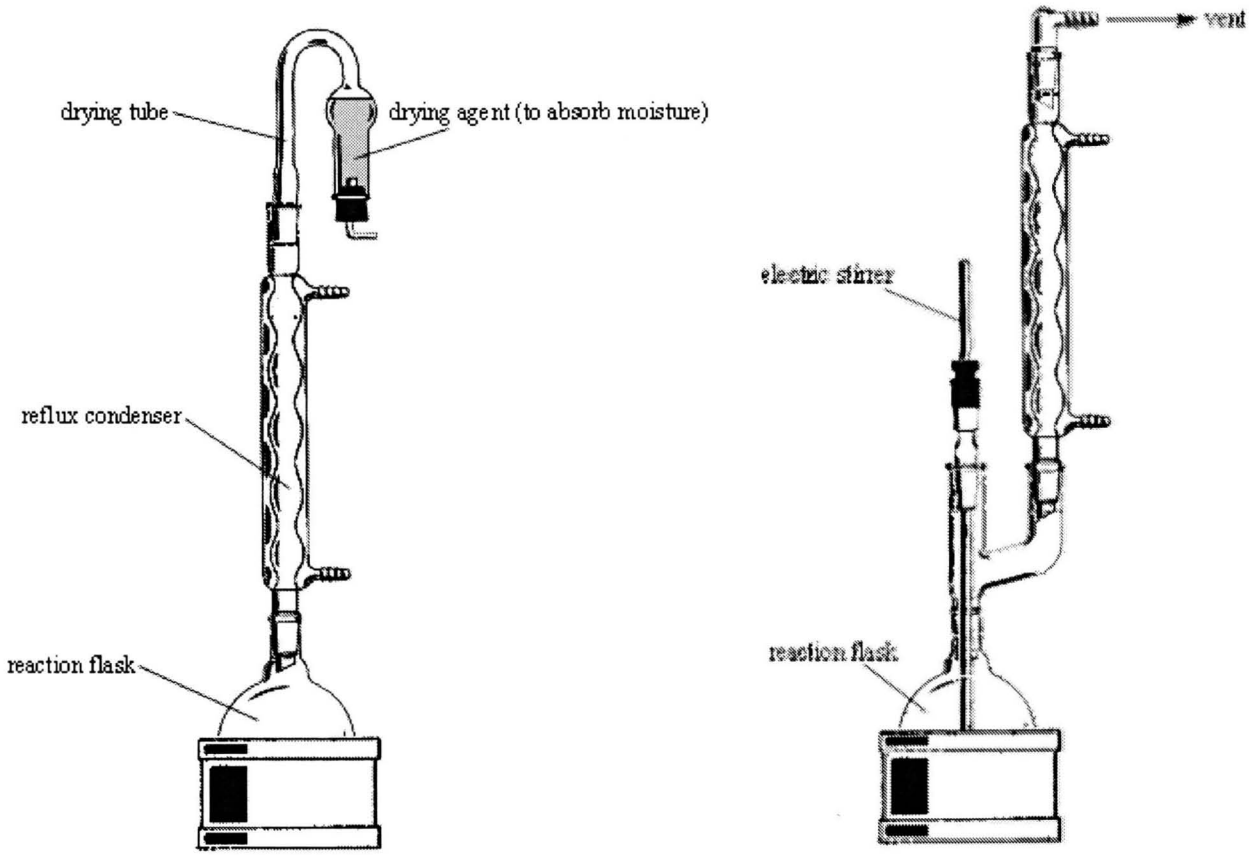


Figure 021. Left illustration: Standard reflux apparatus with drying tube attached to the reflux condenser to exclude moisture. Right illustration: Standard reflux apparatus with motorized stirrer. The vent can be secured to some rubber tubing, which can be submerged into a bath of sodium carbonate for neutralizing acid vapors, for example.

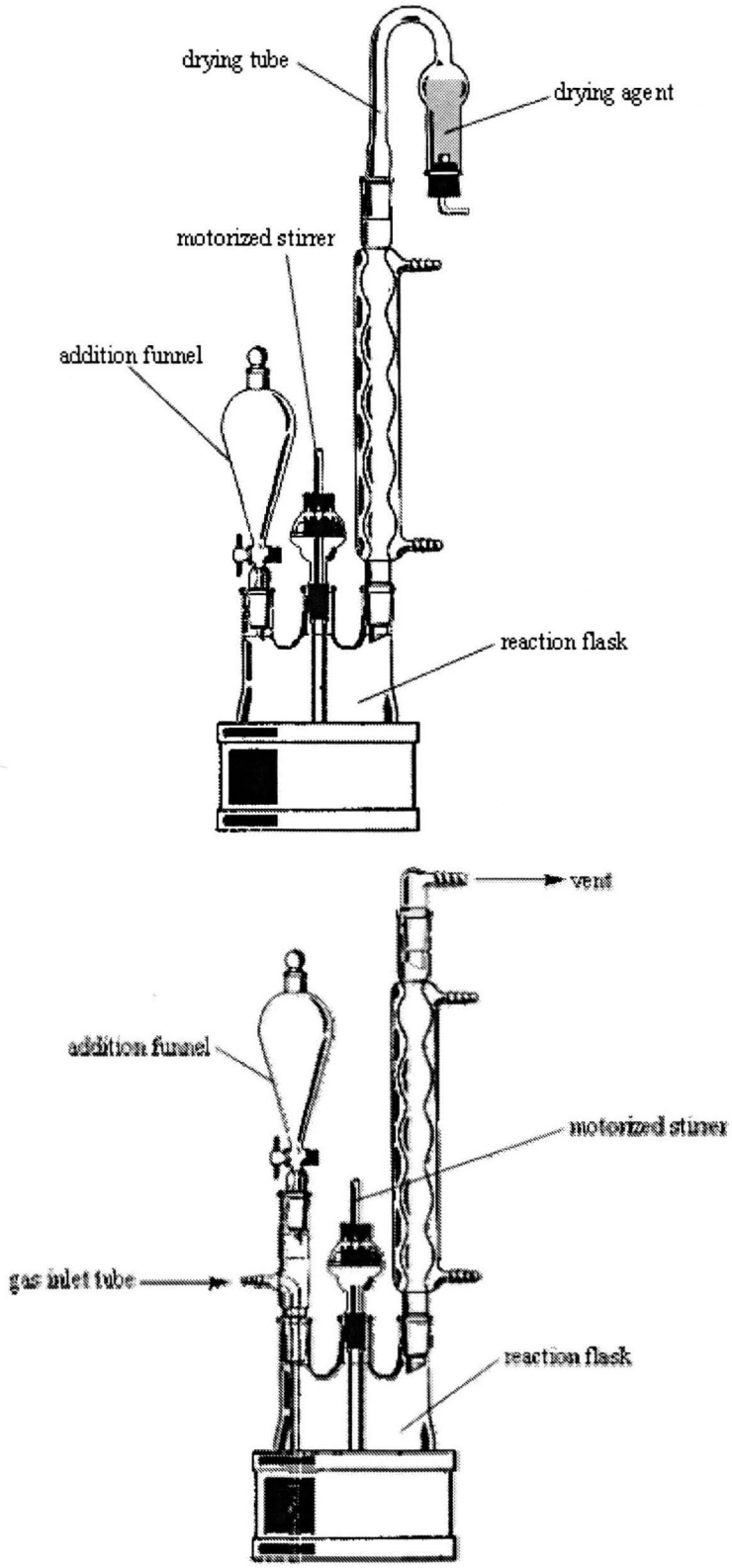


Figure 022. Left illustration: A reflux apparatus with motorized stirrer and addition funnel. The drying tube is attached to the reflux condenser to absorb moisture. Right illustration: A similar apparatus but with gas inlet tube and vent. The vent can be attached to rubber tubing, which can be submerged in a sodium carbonate bath to absorb acidic vapors, for example.

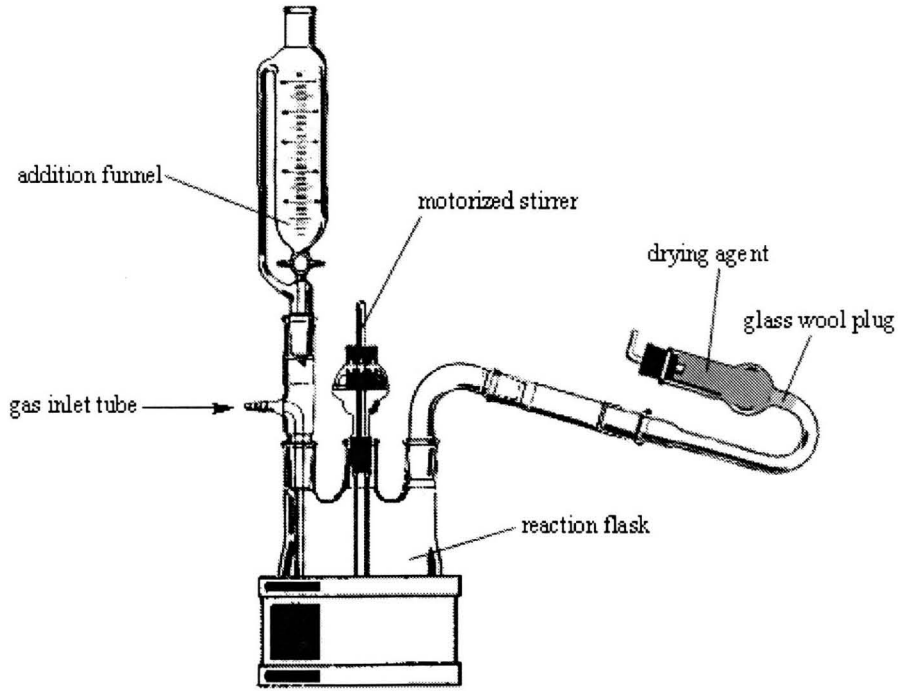


Figure 023. Reaction apparatus with gas inlet tube, addition funnel, and motorized stirrer.

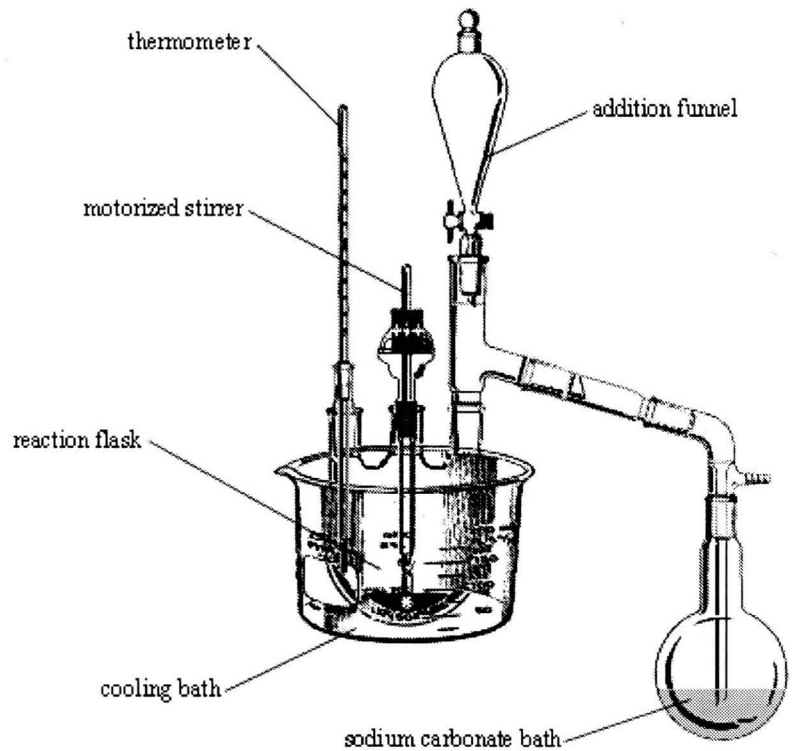


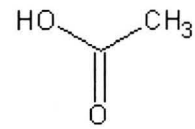
Figure 024. Apparatus with cooling bath, thermometer, motorized stirrer and addition funnel.

Note: All the glassware represented in the illustrations can be purchased from *Lab Glass Inc. PO Box 688, Buena, NJ 08310 USA*. Lab Glass is by far one of the best manufacturers and suppliers of laboratory glassware in North America.

SECTION 3: REFERENCE GUIDE

Intermediates, Reagents, and Solvents used in section 3

Acetic acid, Glacial. *Pure acetic acid*

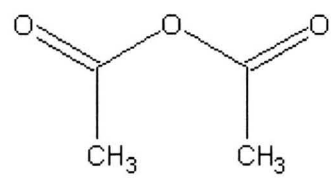


Glacial acetic acid is a colorless liquid with a pungent odor. Glacial acetic acid is a corrosive liquid with a boiling point of 118 Celsius, and a melting point of 17 Celsius. Glacial acetic acid is a flammable liquid. It is an excellent solvent for many organic compounds, and is miscible with water, alcohol, glycerol, ether, and carbon tetrachloride. Glacial acetic acid weekly ionizes in water solutions. It can be made by the destructive distillation of wood, and subsequent condensation of the vapors (methanol is a by-product and hence must be separated by distillation). Glacial acetic acid is a common commercially available chemical. *Wear gloves when handling glacial acetic acid because it can cause skin irritation and possible skin burns.*

15% Acetic acid solution: Prepare by diluting 100 grams of glacial acetic acid into 560 milliliters of water.

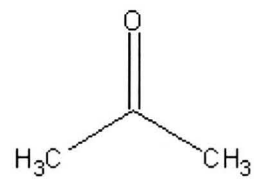
10% Acetic acid solution: Prepare by diluting 50 grams of glacial acetic acid into 450 milliliters of water.

Acetic anhydride



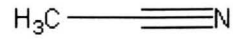
Acetic anhydride is a colorless liquid with a melting point of –73 Celsius, and a boiling point of 139 Celsius. The liquid is corrosive and has a strong acetic acid odor. When mixed with water, it slowly decomposes into acetic acid, and it reacts with alcohols forming acetates. Acetic anhydride is soluble in chloroform and ether. It is made by reacting sodium acetate with acetyl chloride or sulfuryl chloride. Acetic anhydride may produce irritation of the skin, so wear gloves when handling.

Acetone



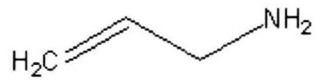
Acetone is a very volatile, highly flammable liquid with a rather characteristic odor, and a pungent, sweetish taste. It has a boiling point of 56 Celsius and a melting point of –94 Celsius. Acetone is miscible with water, alcohol, chloroform, ether, and most oils. Acetone will dissolve many plastics, and resins. Keep acetone away from plastic eyeglass frames, jewelry, pens and pencils, rayon stockings, and other rayon garments. It is mildly irritating, and fumes produce dizziness, and headaches. Use proper ventilation when handling acetone. Acetone is prepared by the oxidation of isopropyl alcohol with potassium dichromate, and on an industrial scale by the oxidation of cumene with air. Acetone is a very common and widely available solvent found in any hardware store.

Acetonitrile



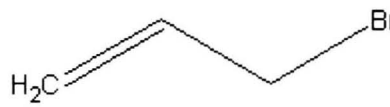
Acetonitrile is a colorless liquid with an ether-like odor. Acetonitrile is a toxic liquid, and can be absorbed through the skin. It is flammable burning with a luminous flame. It has a melting point of -45 Celsius, and a boiling point of 82 Celsius. It is miscible with water, methanol, methyl acetate, ether, chloroform, carbon tetrachloride, and many unsaturated hydrocarbons. Acetonitrile is an excellent solvent that dissolves many inorganic salts. It forms a constant boiling mixture with water (84% acetonitrile with a boiling point of 76 Celsius). Acetonitrile occurs in coal tar, and is a widely available commercial chemical. *Wear gloves and use proper ventilation when handling acetonitrile. Acetonitrile is toxic by inhalation or skin absorption.*

Allylamine



Allylamine forms a colorless liquid with an irritating and strong ammonia like odor. It has a boiling point of58 Celsius. The liquid is soluble in water, alcohol, chloroform, and ether. Use proper ventilation when handling this compound. Allylamine is prepared in a two-step process, starting with the formation of allyl chloride. Allyl chloride is prepared by carefully reacting chlorine with propylene gas. The allyl chloride is then converted into allylamine by reaction with ammonia gas, followed by treating the reaction mixture with sodium carbonate, and then distilling.

Ally bromide



Ally bromide forms a colorless liquid with an unpleasant and pungent odor. It has a melting point of –119 Celsius, and a boiling point of 71 Celsius. The liquid is slightly soluble in water but readily soluble in alcohol, chloroform, ether, and carbon disulfide. It can be made by reacting hydrobromic acid with ally alcohol, and then collecting the ally bromide by fractional distillation using a two-path system.

Aluminum

Al

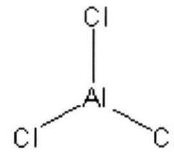
Aluminum is a whitish to silvery white metal with a dull sheen. Aluminum is capable however, of producing a brilliant sheen, which is seen in aluminum foil. It has a melting point of 660 Celsius, and a boiling point of 2327 Celsius. Aluminum foil cannot be properly melted as it forms a layer of aluminum oxide. Aluminum readily reacts with acids, and bases, especially hydrochloric acid and sodium hydroxide. The metal is readily available in the form of nails, rods, and foil.

Aluminum bromide

$AlBr_3$

Aluminum bromide forms white to yellowish-red crystals, powder, or lumps. The compound has a melting point of 97 Celsius, but may have a melting point ranging from 250 to 270 Celsius. The compound fumes in air, forming hydrogen bromide and aluminum oxide, and it reacts violently with water. Store aluminum bromide in airtight containers in a cool dry place. It is soluble in many organic solvents. Aluminum bromide forms the hexahydrate on prolonged storage. It can be made by reacting aluminum foil pieces with a 5% bromine in dry chloroform solution, followed by evaporation of the solvent

Aluminum chloride, Anhydrous



Anhydrous aluminum chloride is white when pure, but is ordinarily a gray or yellowish powder, which fumes in air, and has a strong hydrogen chloride odor. Anhydrous aluminum chloride volatizes when heated in small quantities. It combines with water with explosive violence and liberation of much heat. Anhydrous aluminum chloride is very soluble in most organic solvents. Anhydrous aluminum chloride is a powerful catalyst for the halogenation of organic compounds. It is prepared by passing dry hydrogen chloride gas over finely divided aluminum.

Ammonia, anhydrous

NH_3

Anhydrous ammonia is a colorless gas with a very pungent odor. It has a melting point of -77 Celsius, and a boiling point of -33 Celsius. Ammonia is regarded as nonflammable, but mixtures with oxygen can ignite. Anhydrous ammonia is a corrosive alkaline gas that is very soluble in water. It is also soluble in alcohol, chloroform, and ether. Liquid ammonia is a good solvent for many elements and compounds. Commercial anhydrous ammonia is supplied in the form of a compressed gas in steel tanks, or in the liquid form supplied in steel tanks. Ammonia is also widely sold in water solutions. Anhydrous ammonia is the 4th largest chemical produced in the US. Anhydrous ammonia is a widely available commercial chemical. Anhydrous ammonia is prepared on an industrial scale from hydrogen and nitrogen at high pressure and temperature in the presence of platinum. The average ammonia plant produces 1000 tons of liquid ammonia per day.

Method of preparing anhydrous ammonia

Summary: Anhydrous ammonia can be prepared by treating an ammonium salt solution with a solution of sodium hydroxide. In the following procedure, ammonium chloride or ammonium sulfate is treated with sodium hydroxide to yield ammonia gas, which is then dried to yield the anhydrous gas. The ammonium chloride or sulfate can be prepared by neutralizing 10% ammonia (store bought ammonia) with hydrochloric or sulfuric acids, followed by recrystallization.

Hazards: Wear gloves when handling 50% sodium hydroxide. The alkaline solution may cause painful skin burns on prolonged exposure, and itching sensation on short exposure. Use proper ventilation when making ammonia gas, and avoid inhalation of the vapors.

Procedure: Place 156 grams of 10% ammonia into a flask and then rapidly add 100 grams of 35% hydrochloric acid, or 47 grams of 98% sulfuric acid while stirring the 10% ammonia solution. After the addition of the acid, continue stirring for ten minutes, and then recrystallize the ammonium salt from solution. Afterwards, place the dry recrystallized ammonium salt into an apparatus as illustrated below, and then prepare a sodium hydroxide solution by dissolving 38 grams of sodium hydroxide into 38 milliliters of water (much heat is produced when dissolving sodium hydroxide into water). After the sodium hydroxide solution has cooled to room temperature, add it the ammonium salt drop-wise over a period of about forty minutes. During the addition of the sodium hydroxide, ammonia gas will be steadily evolved.

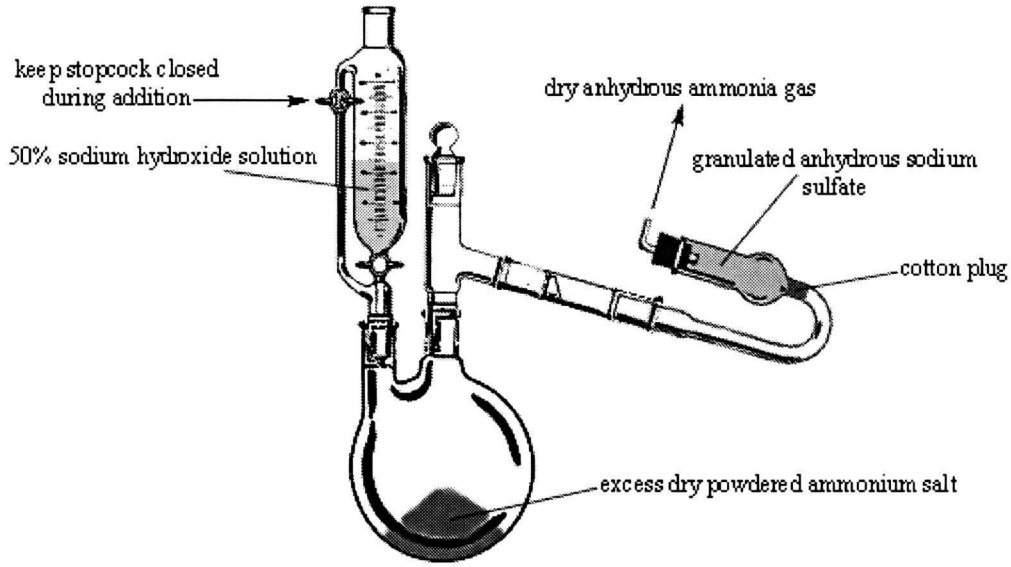


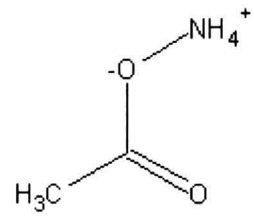
Figure 025. Apparatus for the preparation of anhydrous ammonia.

28 - 30% ammonia solution

28- 30% Ammonia can be prepared by dissolving 20 grams of ammonia gas into 48 milliliters of water. 28 – 30% ammonia is a highly irritating liquid, which evolves highly irritating vapors. Use proper ventilation when handling this substance. 28 – 30% ammonia solution is commercially available, but can be obtained by bubbling ammonia gas into a minimal amount of ice water, or by carefully distilling dilute aqueous mixtures.

10% Ammonia solution: prepare by diluting 350 grams of 28 to 30% ammonia into 750 milliliters of water. Note: 10% ammonia is the common store bought stuff sold as "crystal clear ammonia", or other brands which are colorless.

Ammonium acetate, anhydrous



Ammonium acetate forms colorless to white crystals or powder with a melting point of 114 Celsius. The crystals are somewhat unstable, and tend to decompose loosing ammonia over time. The compound is readily soluble in water and alcohol, but only slightly soluble in acetone. Ammonium acetate can be prepared by reacting ammonia gas with glacial acetic acid, and then recrystallizing the ammonium salt there from.

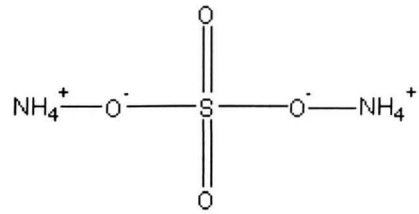
Ammonium chloride. *Ammonium muriate; Sal ammoniac; Amchlor*

$$NH_4Cl$$

Ammonium chloride forms colorless, odorless crystals or crystalline masses, or white granular powder. It has a cooling saline taste, and is mildly hygroscopic. It tends to cake on standing, and sublimes without melting. Ammonium chloride is soluble in water, methanol, and ethanol, but insoluble in acetone, ether, and ethyl acetate. It liberates ammonia gas when treated with alkali solutions, and is prepared by neutralizing hydrochloric acid with ammonia water, followed by recrystallization. It is a widely available commercial chemical.

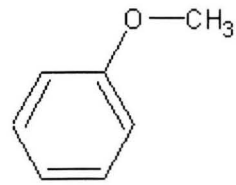
22% Ammonium chloride solution: Prepare by dissolving 50 grams of ammonium chloride into 175 milliliters of water.

Ammonium sulfate



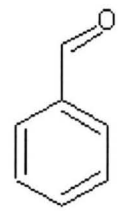
Ammonium sulfate forms odorless, orthorhombic crystals or white granules, which decompose when heated above 280 Celsius. The ammonium salt is soluble in water, but insoluble in alcohol, and acetone. It is easily prepared by neutralizing dilute sulfuric acid with ammonia water, and then recrystallizing the salt.

Anisole



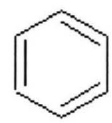
Anisole is a liquid with an agreeable aromatic odor, and a melting point of -37 Celsius. It has a boiling point of 156 Celsius. It is soluble in alcohol and ether, but insoluble in water. Anisole can be prepared from dimethyl sulfate and phenol, and can also be prepared from phenol, methyl iodide, and potassium carbonate in dimethylformamide. Anisole is commercially available, and occurs in some medicated creams.

Benzaldehyde



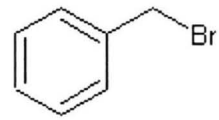
Benzaldehyde forms a colorless to slightly yellowish liquid with a melting point of –57 Celsius, and a boiling point of 179 Celsius. The liquid is insoluble in water, but miscible with alcohol, ether, and most oils. Benzaldehyde occurs in the kernels of bitter almonds, and can be obtained by steam distillation of these kernels. Benzaldehyde is made in the lab by reacting benzyl dichloride (benzal chloride) with lime.

Benzene



Benzene is a clear, colorless, volatile, highly flammable liquid. Benzene is toxic, and a carcinogen. It has a boiling point of 80 Celsius, and is miscible with alcohol, chloroform, ether, carbon disulfide, and carbon tetrachloride. It is insoluble in water. Keep benzene in tightly sealed glass bottles, and store in a cool area. Benzene is a common well-known solvent available commercially, but purchasing may be difficult due its EPA regulation. *Use proper ventilation when handling benzene, as inhalation of benzene vapors are hazardous.*

Benzyl bromide



Benzyl bromide forms a colorless liquid with a strong irritating and characteristic odor. It has a melting point of –4 Celsius, and a boiling point of 200 Celsius. It is soluble in alcohol, chloroform, and ether. Benzyl bromide should be kept away from iron, which causes it rapid decomposition; as well does water but only slowly. Benzyl bromide can be made by mixing an excess of a solution of bromine in methylene chloride with toluene, and exposing the combined mixture to direct sunlight for several hours.

Bromine

Br_2

Bromine is a dark red, highly fuming liquid, which is very volatile. Its fumes are toxic, corrosive, and strongly irritating. Bromine has a melting point of –7 Celsius, and a boiling point of 59 Celsius. It is insoluble in water, but freely soluble in alcohol, ether, chloroform, and carbon disulfide. It is soluble in alkali bromide solutions. Bromine is less reactive then chlorine, but just as toxic. Keep bromine stored in glass stoppered bottles, and store in a cool place (refrigerator) away from sunlight. Bromine is prepared by passing chlorine gas into a solution of sodium bromide, and then simultaneously evaporating the bromine. The bromine vapors are then condensed. Bromine is commercially available but shipping regulations may restrict its sale.

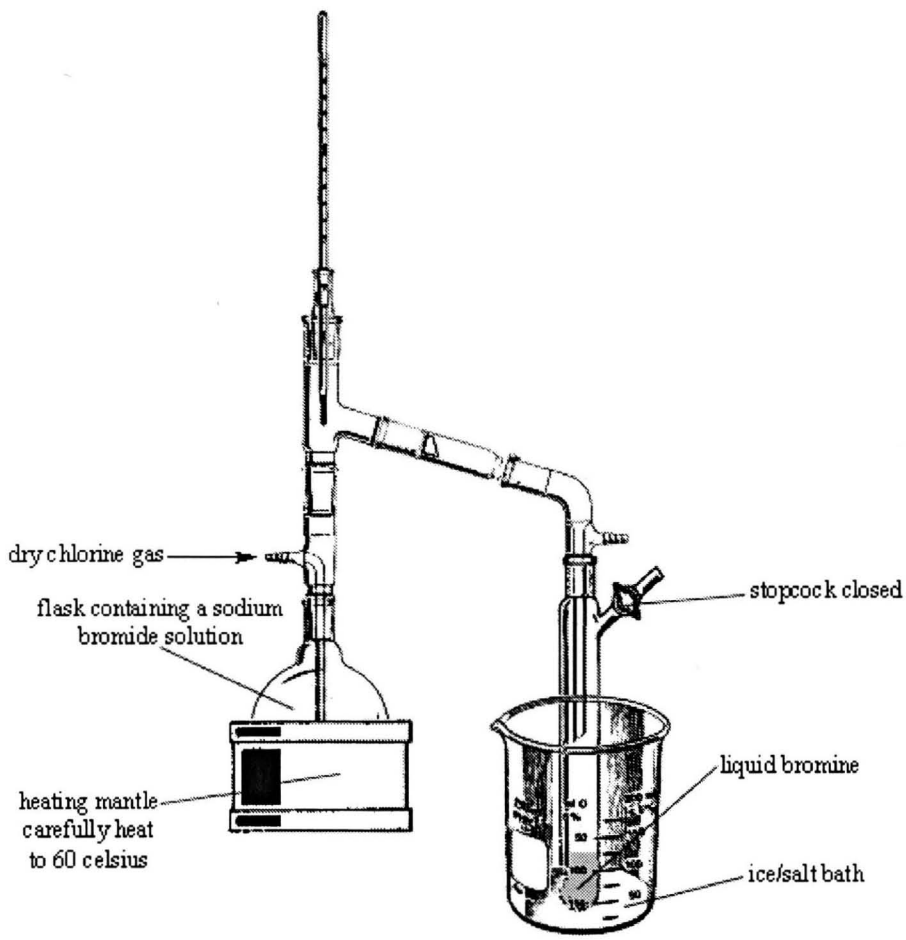


Figure 026. Apparatus for preparing bromine. The bromine should be re-distilled at 60 Celsius.

1-Bromopropane

Bromopropane forms a colorless to lightly yellowish liquid. It can be made by refluxing a mixture of 1-propanol with potassium or sodium bromide in the presence of 50% sulfuric acid, and then distilling-off the bromopropane.

n-Butylamine

n-Butylamine forms a colorless oily liquid with an ammonia like odor. It has a melting point of –50 Celsius, and a boiling point of 78 Celsius. The liquid is miscible with water, alcohol, and ether. N-Butylamine is manufactured on an industrial scale from butanol and ammonia, but can be made in the lab by reacting n-butyl chloride with excess ammonia gas in ether at 0 Celsius, filtering-off the precipitated hydrochloride, dissolving this hydrochloride into water, then adding in a sodium carbonate solution, and then distilling out the n-butylamine.

tert-Butyl lithium

tert-Butyl lithium is a highly reactive substance capable of igniting in the air. It should be stored in ether, submerged in kerosene, or wet with tetrahydrofuran until use. It can be made by adding metallic lithium to a pure dry solution of tert-butyl chloride in ether, filtering-off the lithium chloride, and then removing the ether.

Calcium bicarbonate

$$Ca(HCO_3)_2$$

Calcium bicarbonate forms a white powder, lumps, or granules, which are insoluble in water, but readily soluble in acids forming the calcium salts. Calcium bicarbonate can be prepared by bubbling carbon dioxide gas into a suspension of calcium hydroxide in water, or by treating a suspension of calcium hydroxide in water with dry ice, followed by filtration. The filtered-off product will still contain some calcium hydroxide.

Calcium hypochlorite

$$Ca(OCl)_2$$

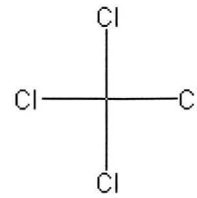
Calcium hypochlorite forms yellowish to light yellowish gray powder with a peculiar chlorine-like odor. Calcium hypochlorite has never been obtained in pure state, and only exists in admixture with calcium hydroxide, lime, and calcium chloride in the form of "bleaching powder", which usually contains about 70% calcium hypochlorite by weight (65% available chlorine). Calcium hypochlorite is made by chlorinating lime, and is widely available as a pool chemical.

Calcium oxide

$$CaO$$

Calcium oxide forms a white to grayish white powder, lumps, or granules. The compound readily absorbs carbon dioxide and moisture from the air. Calcium oxide is prepared by roasting calcium hydroxide or carbonate for several hours at high temperature. The calcium hydroxide can be prepared by treating calcium chloride with sodium hydroxide solution, filtering-off the calcium hydroxide, and then roasting said compound.

Carbon tetrachloride



Carbon tetrachloride is colorless, heavy, non-flammable liquid with a characteristic odor. It has a boiling point of 78 Celsius, and a melting point of –23 Celsius. Carbon tetrachloride is insoluble in water, but miscible with alcohol, benzene, chloroform, ether, and carbon disulfide. Carbon tetrachloride is a potential poison, and inhalation, ingestion, and skin absorption should be avoided at all cost. Carbon tetrachloride may be a carcinogen. It is prepared on an industrial scale by the chlorination of methane, but can be conveniently prepared by reacting chlorine with carbon disulfide in the presence of iron fillings; the carbon tetrachloride is recovered by distillation.

Cesium carbonate

$$Cs_2CO_3$$

Cesium carbonate forms crystals, powder, or granules, which melt only when strongly heated. The crystals are very soluble in water, and soluble in alcohol and ether. Cesium carbonate is commercially available, but expensive. It can be made by roasting cesium oxide with magnesium carbonate, then leaching out the cesium carbonate with water, filtering, and then recrystallization.

Chlorine gas

$$Cl_2$$

Chlorine gas is a yellow gas with a suffocating, and strongly irritating odor. It has a melting point of –101 Celsius, and a boiling point of –34 Celsius. Chlorine is sold as a compressed gas in steel cylinders. It is insoluble in water and not very soluble in alcohol, but soluble in dry benzene, and toluene. Chlorine combines readily with all elements except the noble gases, hydrogen, oxygen, and nitrogen. Chlorine does not occur naturally, but occurs in combined form as chlorides. It occurs in nature (in the form of chlorides) as sodium chloride, potassium chloride, and magnesium chloride. Many finely divide metals will burn in a chlorine atmosphere. Chlorine is a toxic gas, which can be fatal if inhaled for prolonged periods of time. Inhalation of mild quantities of chlorine causes nose and throat irritation followed by excessive mucous congestion in the nose. Chlorine is a corrosive gas, which will react with many metals on contact. It is a strong oxidizer and is capable of oxidizing a great many inorganic compounds. Chlorine will explodes in contact with hydrogen if direct sunlight is present. Chlorine should be protected from sunlight. It is the 10^{th} largest chemical manufactured in the US. It was used as a chemical warfare agent in WWI, but due to its lack of toxicity, and poor environmental capacity (dissipates rapidly), its use has ended. Chlorine is prepared on an industrial scale from the electrolyses of sodium chloride brine in a system called the chloro-alkali process (sodium hydroxide is a useful by-product). It can be prepared in the lab by reacting hydrochloric acid with calcium hypochlorite or liquid bleach (Clorox), by the electrolysis of hydrochloric acid, or by using a diaphragm cell. Note: In the electrolysis of hydrochloric acid, hydrogen gas is also produced. Despite the presence of this hydrogen, the chlorine can be directly used in chemical reactions because the hydrogen acts more like an inert gas. The chlorine does not react with this hydrogen, even if the gas mixture is moderately hot.

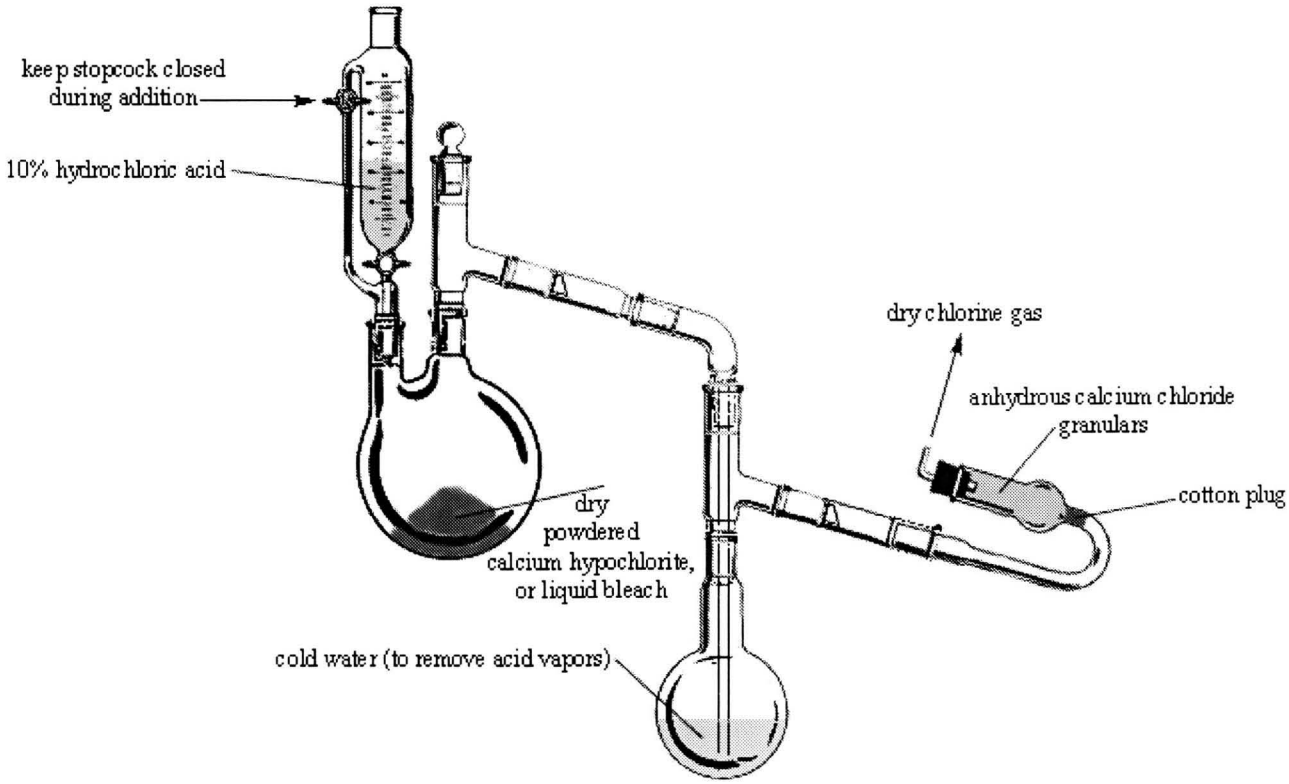


Figure 027. Apparatus for preparing chlorine gas (manganese dioxide, or potassium permanganate can be used instead of calcium hypochlorite).

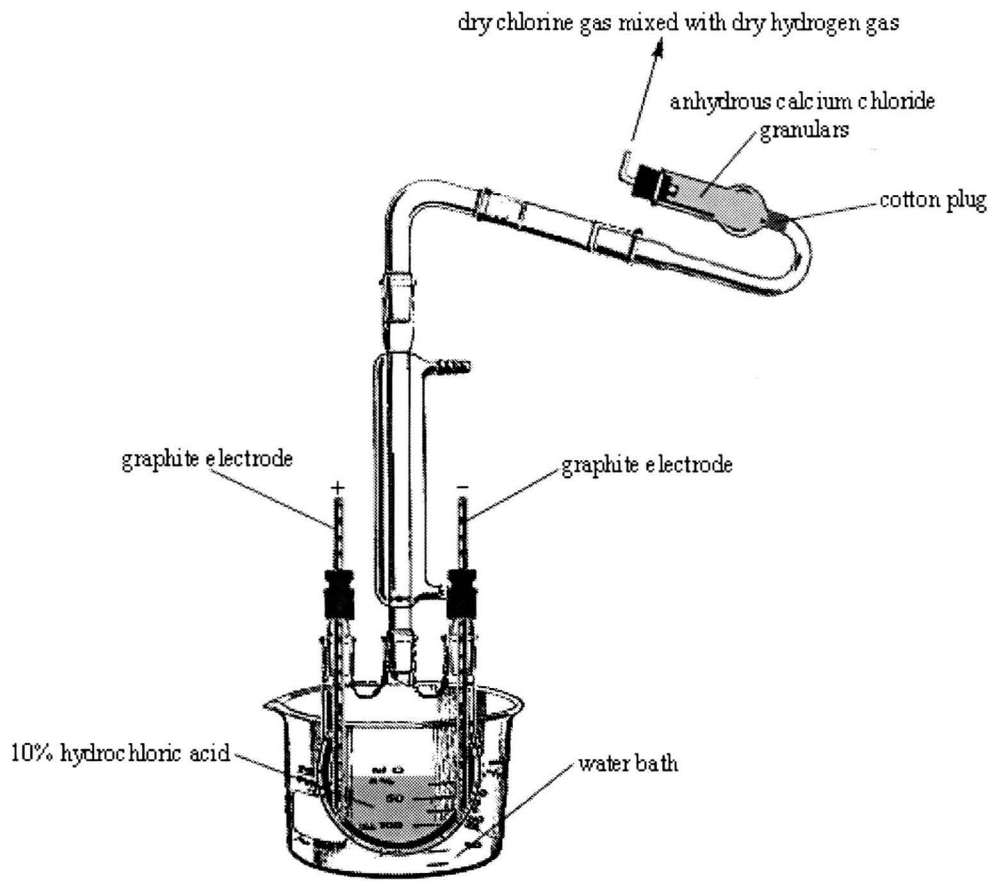


Figure 028. Apparatus for the electrolysis of hydrochloric acid. Warning: If the apparatus is exposed to direct sunlight, detonation will occur. Chlorine explodes when mixed with hydrogen and exposed to sunlight. The detonation propagates downwards, so there is no immediate danger from fragments. As a reminder, the electrolysis of hydrochloric acid is perfectly safe as long as the apparatus is protected from direct sunlight, magnesium light, halogen lamps, or UV lamps (cover all windows, ect.).

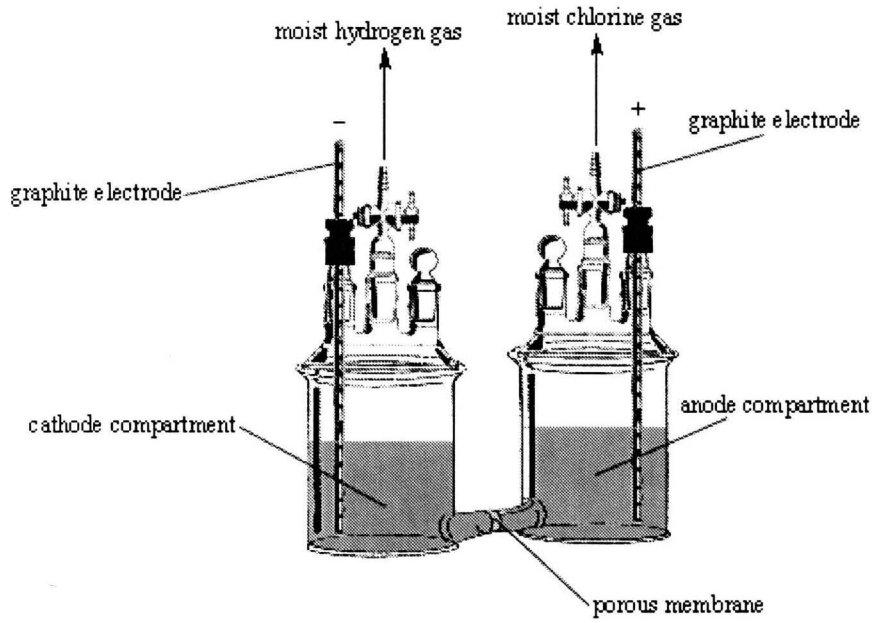
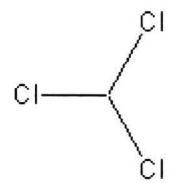


Figure 029. Diaphragm cell for the production of chlorine. The moist chlorine can be easily dried using a calcium chloride drying tube, and afterwards, will be in high purity.

Chloroform. *Trichlormethane*



Chloroform is a highly refractive, nonflammable, heavy, very volatile, and sweet-tasting liquid with a peculiar odor. It has a boiling point of 62 Celsius, and a melting point of -64 Celsius. Chloroform forms a constant boiling mixture with alcohol containing 7% alcohol, and boiling at 59 Celsius. Commercial chloroform contains a very small amount of ethanol as stabilizer. It is insoluble in water, but miscible with alcohol, benzene, ether, petroleum ether, and carbon disulfide. Pure chloroform is light sensitive, so store in amber glass bottles in cool place. Chloroform is a suspected light carcinogen, so use proper ventilation when handling. Over exposure to chloroform vapors causes dizziness, and headache. **Note:** Distilling mixtures containing chloroform mixed with one or more strong base (lithium, sodium, or potassium hydroxide) can result in explosion. Always neutralize any base, or extract the chloroform before distilling the chloroform.

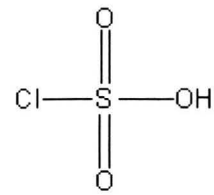
Method of preparing chloroform

Summary: Chloroform is prepared by reacting acetone with calcium hypochlorite, and then extracting the mixture with toluene. After extraction, the toluene/chloroform mixture is then double distilled to collect the chloroform in the receiver flask. After collecting the chloroform, it is mixed with a small amount of 95% ethanol to act as a stabilizing agent.

Hazards: Extinguish al flames before suing acetone, which is highly volatile and flammable. Calcium hypochlorite is a powerful oxidizer, and should never be mixed with concreted sulfuric acid; explosions will result. Chloroform inhalation should be avoided, but is not threatening in mild condition.

Procedure: Place 100 grams of water and 100 grams of acetone into a beaker, and then cool to 0 Celsius while stirring. Then slowly add in small portions, 1181 grams of 65 to 70% calcium hypochlorite (commercially available; sold under a variety of brand names for use in swimming pools and hot tubs) over a period of 1 hour while stirring the acetone solution and maintaining its temperature at 0 Celsius. After the addition of the 65 to 70% calcium hypochlorite, continue to stir the reaction mixture at 0 Celsius for an additional thirty minutes. Afterwards, stop stirring and then extract the reaction mixture with four 100-milliliter portions of toluene. After extraction, combine all four portions (if not already done so), and then place the combined portions into a distillation apparatus and then distill at 65 Celsius until no more chloroform passes into the receiver flask. When no more chloroform passes into the receiver flask, stop the distillation, and then remove the receiver flask from the distillation apparatus. Then add 20 grams of anhydrous calcium chloride to the receiver flask, and then swirl the flask for ten minutes. After which, filter-off the calcium chloride and then pour the filtered chloroform into a clean distillation apparatus and distill at 62 Celsius until no more chloroform passes into the receiver flask. When no more chloroform passes into the receiver flask, stop the distillation, and then remove the chloroform from the receiver flask and then add 1 milliliter of 95% ethanol to the chloroform. Then store the chloroform in an amber glass bottle.

Chlorosulfonic acid



Chlorosulfonic acid forms a colorless to slightly yellow liquid, with a melting point of –80 Celsius, and a boiling point of 155 Celsius. The liquid is very corrosive and is capable of producing skin burns—wear gloves when handling and avoid inhalation or ingestion. The liquid fumes in air and has a pungent odor. Keep chlorosulfonic acid out of contact with water, as it reacts with explosive violence forming hydrogen chloride and sulfuric acid. Chlorosulfonic acid is prepared by mixing dry hydrogen chloride gas with dry sulfur trioxide at 50 Celsius in a large reaction flask (fitted with a vertical cold water condenser), and allowing the vapors of chlorosulfonic acid to condense.

Copper

$$Cu$$

Copper is a very common metal that occurs in the form of wires, pipes, tubes, and many other shapes and sizes. It has a familiar and characteristic color, with a metallic sheen. It is stable in air at ordinary temperature but tends to blacken on exposure to high temperatures. The metal is readily available from any hardware store.

Copper-II-sulfate. *Cupric sulfate*

$$CuSO_4$$

Copper-II-sulfate occurs in nature as the mineral ***hydrocyanite.*** The anhydrous salt forms grayish-white to greenish-white rhombic crystals or amorphous powder, which decompose above 560 Celsius. The anhydrous salt is hygroscopic. It is soluble in water and alcohol. The anhydrous salt is prepared by heating the hydrates to 250 Celsius for 1 hour. The monohydrate is a hygroscopic white powder, which is soluble in water, but insoluble in alcohol. The Pentahydrate occurs in nature as the mineral ***chalcanthite,*** *and it* forms large, blue or ultramarine, triclinic crystals or blue granules or light-blue powder. It slowly effloresces in air, and is converted to the anhydrous salt by heating to 250 Celsius for 1 hour. It is very soluble in water, methanol, and glycerol. Copper-II-sulfate is highly toxic to algae, moss, and fungus. Copper and its salts are so toxic to algae, moss, and fungus that a single copper strip placed at the top of a shower or bath-tube will keep it mildew free for decades. Copper-II sulfate is commercially available, but can be made by either neutralizing dilute sulfuric acid with copper-II-oxide, followed by recrystallization to obtain the pentahydrate, or by electrolyzing a solution of magnesium sulfate (Epsom salt) using a copper electrode in a two chamber cell. Note: a clay pot can be used by placing a clay pot in side a larger plastic container. The contents in the clay pot act as the cathode compartment, and the contents in the outer container act as the anode compartment, or vise versa.

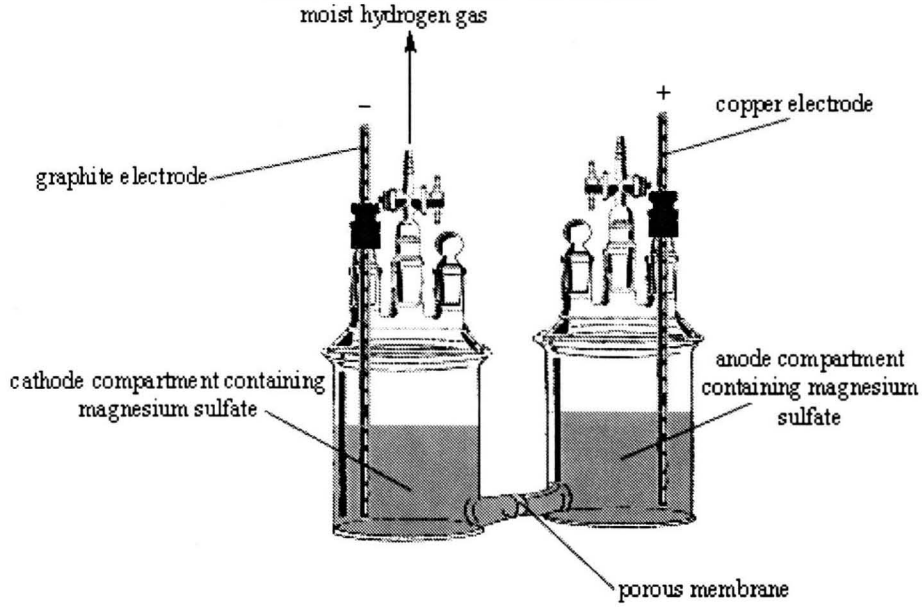
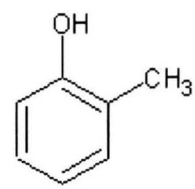


Figure 030. Apparatus for preparing copper-II-sulfate. The copper-II-sulfate forms in the anode compartment and can be collected by recrystallization. Magnesium hydroxide precipitates in the cathode compartment, and is a useful by-product.

Copper zinc alloy (8% copper by weight)

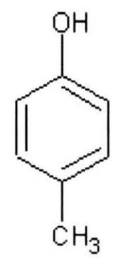
Prepare by heating 110 parts of zinc to 420 Celsius. Alter the zinc has melted, add in 10 parts of copper, and then heat the entire mixture until a uniform molten mix is formed. Thereafter, slowly pour this molten mass into hot water, and then filter-off the insoluble copper/zinc alloy pieces. These pieces can be pulverized into smaller pieces if desired.

ortho-Cresol



ortho-Cresol forms colorless to slightly yellow crystals or liquid, which become dark on standing and on exposure to air and light. The crystals have a phenol like odor with a melting point of 30 Celsius and a boiling point of 192 Celsius. The compound is insoluble in water, but soluble in most solvents.

para-Cresol



para-Cresol forms a colorless to yellowish semi liquid mass with a melting point of 36 Celsius, and a boiling point of 202 Celsius. The compound is volatile with steam. para-Cresol is insoluble in water, but readily soluble in alcohol, ether, and many organic solvents. It can be made by reacting fuming sulfuric acid with toluene, followed by fusing the recovered sulfonate crystals with potassium hydroxide, and then leaching out the desired para-cresol with ether, followed by evaporation of the ether.

Cuprous bromide. Copper-I-bromide

CuBr

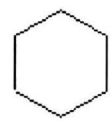
Cuprous bromide forms a white powder, which turns green to blue on exposure to air and sunlight. The powder should be stored in airtight containers in a cool dry place. The compound has a melting point of 504 Celsius. Cuprous bromide is insoluble in water and most solvents. Cuprous bromide is made in a similar manner as cuprous chloride.

Cuprous chloride. Copper-I-chloride

CuCl

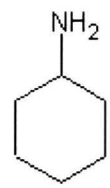
Cuprous chloride forms a white powder, which is stable in air, but turns orange to green when exposed to water. The powder is stable towards cupric chloride solutions and sodium chloride solutions. The powder has a melting point of 430 Celsius. Cuprous chloride is insoluble in water and most solvents. Cuprous chloride can be obtained by electrolyzing a solution of sodium chloride using a two compartment cell, separated by clay, and using a copper anode, and graphite cathode—the chief product is cupric chloride, but a small amount of the cuprous chloride forms in the bottom of the anode compartment. It can also be made by reducing cupric chloride at high temperature with finely divided copper.

Cyclohexane



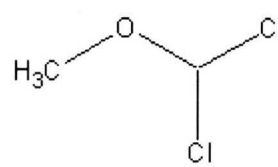
Cyclohexane forms a colorless flammable liquid with a characteristic solvent like odor. It has a melting point of 6 Celsius, and a boiling point of 81 Celsius. Cyclohexane is relatively insoluble in water, but miscible with most common organic solvents. It is a readily available solvent, which can be made by the dehydrogenation of benzene using raney nickel catalyst at 150 Celsius with excess hydrogen under mild pressure.

Cyclohexylamine



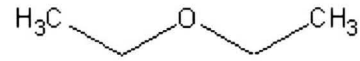
Cyclohexylamine forms a colorless liquid with a fishy ammonia like odor. It has a melting point of –18 Celsius, and a boiling point of 135 Celsius. Cyclohexylamine is miscible with water, alcohols, acetone, ether, hexane, and methylene chloride. It forms an azeotrope mixture with water containing 44% cyclohexylamine by weight with a boiling point of 96 Celsius. The liquid is manufactured by the catalytic hydrogenation of aniline at over 100 Celsius.

Dichloromethyl methyl ether



Dichloromethyl methyl ether is a colorless to yellowish liquid with a strong odor. The liquid is unstable in air and should be stored in tightly sealed bottles in a cool dry place. It can be made by dripping methyl formate into a mixture of phosphorus oxytrichloride and phosphorus pentachlorde at 5 Celsius.

Diethyl ether. *Ether*



Diethyl Ether, other wise known as just ether, is a mobile, very volatile, highly flammable liquid, which produces explosive vapors. It has a sweetish, pungent odor, and a burning taste. Ether forms explosive peroxides when exposed to air—ether containing peroxides will detonate if heated, shattering the glass vessel. Before heating mixtures containing ether, the peroxide test should be conducted. To test for peroxides, add five drops of ferrous chloride solution to the ether mixture. If a red or black color appears, peroxides are present. *Note: This test will not work properly if there are oxidizing agents in the ether mixture.* Ether can be stabilized by the addition of small amounts of naphthols, but this does not protect ether 100% from peroxide formation. Ether has a melting point of –116 Celsius, and a boiling point of 35 Celsius. Ether and air mixtures are explosive, so extinguish all flames and do not smoke when handling it. Protect ether from static electricity, which can also cause fire. Ether is insoluble in water, but miscible with alcohol, benzene, chloroform, and many oils. Do not mix 99% nitric acid with ether, as detonation will take place. Inhalation of ether vapors can produce intoxicating effects. These effects include feelings of euphoria, well-being, relaxation, and a general state of high. These effects can also lead to feelings of drunkness. Ether is a narcotic in high concentrations, but is not habit forming. Store ether in tightly sealed bottles in a cool place (preferably in a refrigerator). For prolonged storage, store ether over sodium sulfite and keep in a bottle filled to the top (to minimize the air space). Ether can be prepared by heating 95% ethanol and 98% sulfuric acid (1 to 1 ratio) to 100 Celsius, and simultaneously condensing the distilled-off vapors of ether. The ether is then purified by re-distillation. Ether is a widely available commercial chemical.

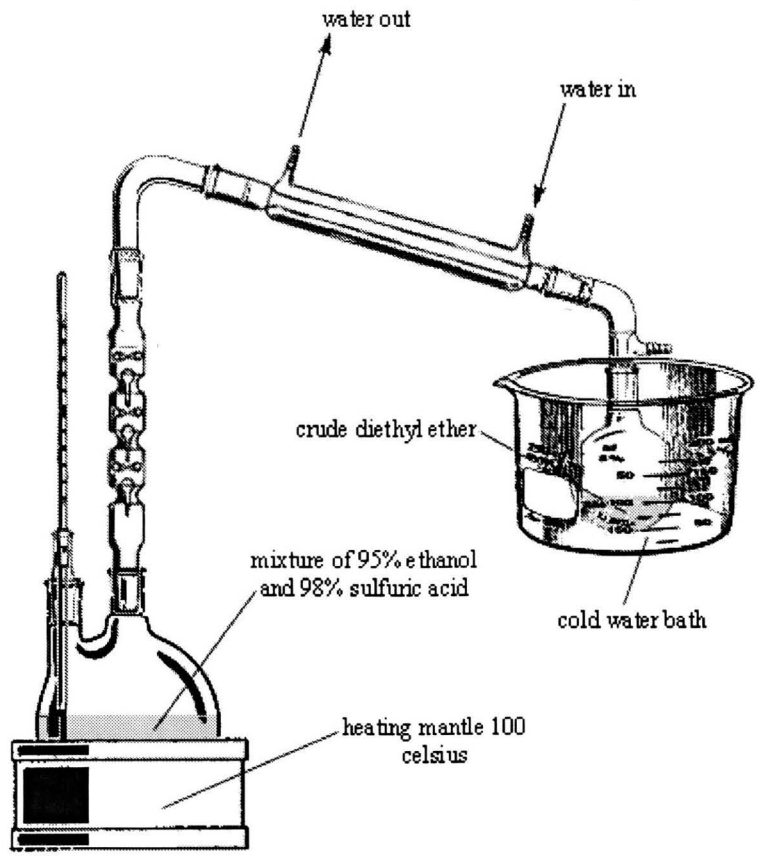
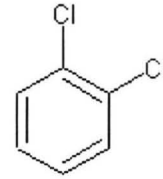


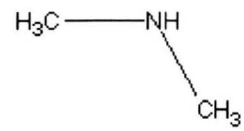
Figure 031. Apparatus for the preparation of diethyl ether. The ether should be re-distilled.

ortho-Dichlorobenzene



ortho-Dichlorobenzene forms a colorless liquid with a melting point of –17 Celsius and a boiling point of 181 Celsius. The liquid is insoluble in water, but miscible with alcohol, ether, and toluene. It can be made by chlorinating benzene at 50 to 60 Celsius, and then separating the isomers using various techniques.

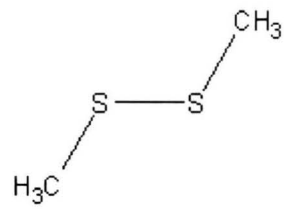
Dimethylamine



Dimethylamine is a colorless gas with a characteristic odor. It has a melting point of –96 Celsius, and a boiling point of 7 Celsius. The gas is very soluble in water, and it aqueous solution is very alkaline. Dimethylamine is usually sold as a gas compressed into cylinders over liquid, or as a 33% aqueous solution. The gas is very soluble in water, and it is appreciably soluble in alcohol and ether. Dimethylamine forms salts with strong acids. It is prepared by passing methanol and ammonia through a copper tube at 200 Celsius, followed by condensation of the resulting vapors, and fractional distillation at low temperature.

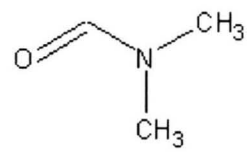
40% Dimethylamine solution: Prepare by dissolving 50 grams of freshly prepared cold liquid dimethylamine into 75 milliliters of ice cold water.

Dimethyldisulfide



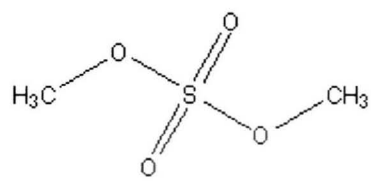
Dimethyldisulfide can be prepared by oxidizing methyl thiol with 30% hydrogen peroxide.

Dimethylformamide (DMF). *N,N-Dimethylformamide*



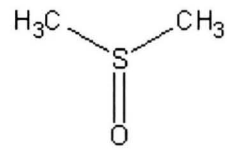
DMF is a colorless, or slightly yellow liquid with a faint amine odor. It has a melting point of -61 Celsius, and a boiling point of 153 Celsius. DMF is miscible with water, and most common organic solvents. It can be prepared from dimethylamine and formic acid. DMF is a common solvent and is commercially available.

Dimethyl sulfate



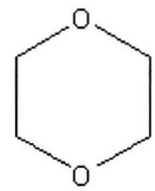
Dimethyl sulfate is a colorless oily liquid with a boiling point of 188 Celsius (with decomposition), and a melting point of –27 Celsius. It is slightly soluble in water, but soluble in ether, dioxane, acetone, and benzene. It is prepared by distilling methanol with fuming sulfuric acid, or by treating dimethyl ether with sulfur trioxide. Dimethyl sulfate is poisonous, and may be a carcinogen so wear gloves, and use proper ventilation when handling. The liquid can be absorbed through the skin.

Dimethylsulfoxide. *Sulfinylbismethane; Methylsulfoxide; DMSO*



Dimethylsulfoxide is a very hygroscopic liquid, which is colorless and odorless. It is has a slightly bitter taste with a sweet after taste. It has a melting point of 18 Celsius, and a boiling point of 189 Celsius. Dimethylsulfoxide is soluble in water, ethanol, acetone, ether, benzene, and chloroform. Keep dimethylsulfoxide in tightly sealed bottles, and away from moisture. It dissolves many gases, and is an excellent solvent. It can be obtained as a by-product in the wood pulp industry, and is commercially available. Due to the high boiling point of dimethylsulfoxide, recovery of it by distillation is not practical. To recover dimethylsulfoxide from reaction mixtures, extract the dimethylsulfoxide with methylene chloride, and then distill-off the methylene chloride to obtain nearly pure dimethylsulfoxide.

Dioxane. *1,4-Diethylene dioxide*

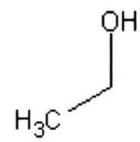


Dioxane is a flammable liquid with a faint pleasant odor. It has a melting point of 12 Celsius, and a boiling point of 101 Celsius. It forms an azeotropic mixture with water containing 81.6% dioxane by weight with a boiling point of 88 Celsius. Dioxane also forms an azeotropic mixture with ethanol containing 9.3% dioxane by weight with a boiling point of 78 Celsius. Dioxane is soluble in water, and the usual organic solvents. It forms explosive peroxides if stored in the presence of air. Dioxane is a toxic carcinogen so avoid ingestion, inhalation, or skin contact. Keep dioxane in tightly sealed bottles filled to the top. Dioxane is prepared by distilling ethylene glycol with dilute sulfuric acid. Dioxane is commercial available, and can be obtained from some solvent mixtures by distillation. Before distilling dioxane that has been in storage for some time, perform the same peroxide test as used with ether.

Procedure for preparing dioxane

Place 100 grams of ethylene glycol and 3.2 grams of 98% sulfuric acid into a distillation apparatus, and then distill the mixture at 110 Celsius for three hours. During the three hours, a distillate containing dioxane, water, and by-products will be obtained in the receiver flask. After the three hours, pour the liquid in the receiver flask into a clean beaker, and then add 60 grams of anhydrous calcium chloride. Then thoroughly blend the mixture for 1 hour. After 1 hour, filter-off the calcium chloride and then place the filtered mixture into a clean distillation apparatus. Then distill at 101 Celsius for 3 hours. After 3 hours, the liquid in the receiver flask will be composed of about 98% dioxane.

95% Ethyl alcohol; *ABS alcohol; grain alcohol*



95% Ethanol is a clear, colorless, very mobile, flammable liquid with a pleasant odor, and a pungent, burning taste. It has a boiling point of 78 Celsius and a melting point of -114 Celsius. 95% Ethanol slowly absorbs water from the air, and dilute ethanol solutions are slowly oxidized by air forming brown colored solutions containing mixtures of aldehydes, and carboxylic acids; mainly acetic acid. 95% Ethanol is miscible with water, and many organic solvents. 95% ethyl alcohol is called absolute

ethanol because ethyl alcohol forms a binary azeotrope containing 95.57% ethyl alcohol by weight with a boiling point of 78 Celsius. Distillations cannot produce 99% ethanol because of this azeotrope. Ethyl alcohol is usually sold as denatured ethyl alcohol (mixed with small amounts of toxic chemicals to make non-drinkable) due to US government tax regulations. 95% Ethyl alcohol is toxic, and ingestion can cause alcohol poisoning. Dilute mixtures of ethanol (Vodka, Gin, Rum, Jack Daniels, beer, wine) produce intoxicating effects when ingested (these intoxicating effects can be increased if the dilute ethanol mixture is injected). 95% ethanol can be made by fermenting starch or sugars with yeast, followed by double distillation. 95% Ethanol is manufactured on an industrial scale by the petroleum industry from ethylene gas, sulfuric acid, and water. 95% Ethanol is a widely available commercial chemical sold under a variety of names. 95% ethanol can be obtained from double distillation of alcoholic beverages such as vodka, gin, or rum.

Method of preparing 95% ethyl alcohol

Summary: 95% ethanol can be prepared by double distilling cheap vodka. After the first distillation the distilled liquid is treated with baking soda to remove odors, filtered, and then the filtered liquid is redistilled producing 95% ethanol.

Procedure: Place 2 liters of cheap vodka (Popov, Kirov, Skol) into a distillation apparatus, and distill at 90 Celsius until no more liquid passes into the receiver flask. When no more liquid passes into the receiver flask, remove the heat source, and then remove the receiver flask from the distillation apparatus. Then place 100 grams of baking soda into the receiver flask, and swirl the flask for ten minutes. Afterwards, filter the liquid to remove the baking soda, and then place the filtered liquid into a clean distillation apparatus. Then distill at 80 Celsius until no more liquid passes into the receiver flask.

Alternative method of preparing 95% ethyl alcohol

Summary: 95% Ethanol can also be obtained on a lower yield by hydrolyzing table sugar with dilute acid, and then fermenting the resulting mixture with yeast to form an ethanol solution. The solution will be contaminated heavily with by-products so multiple distillations and treatments with baking soda will be needed in order to fulfill proper purification. Baking soda is mixed with the distilled liquid to absorb odors and the like.

Procedure: Dissolve 1 kilogram of table sugar (sucrose) into 3 liters of water. Then rapidly stir this sugar mixture, and heat it to 80 Celsius. When the sugar solution reaches about 80 Celsius, continue stirring and add 5 drops of concentrated hydrochloric acid or 5 drops of concentrated sulfuric acid, and then continue heating and stirring for thirty minutes. After thirty minutes, remove the heat source, and allow the mixture to cool to room temperature. Then add 5 grams of baking soda to neutralize the acid. Afterwards, pour the sugar solution into an empty bottle (such as a clean empty plastic milk jug), and then add 5 to 10 grams of regular yeast (bakers yeast or preferably brewers yeast). Then stir the mixture for several minutes to insure good dispersion of the yeast. Then plug the bottles opening with cotton, and then place the bottle into a cool place away from light. Then allow the sugar mixture to ferment for about 4 weeks. After 4 weeks, remove the cotton from the bottles opening, and then pour the contents of the bottle into a distillation apparatus. Then distill at 100 Celsius for 4 ½ hours. After which, remove the heat source, and then remove the receiving flask from the distillation apparatus. Then add 100 grams of baking soda to the contents in the receiving flask, and then swirl the flask for ten minutes. Afterwards, filter the mixture to remove the baking soda, and then place the filtered liquid into a clean distillation apparatus and distill at 80 Celsius until no more liquid passes into the receiver flask. When no more liquid passes into the receiver flask, remove the heat source, and then remove the receiver flask from the distillation apparatus. Then add 100 grams of baking soda to the receiver flask and then swirl the flask for ten minutes. After ten minutes, filter the mixture to remove the baking soda, and then place the filtered mixture into a clean fractional distillation apparatus and distill at 78 Celsius until more ethyl alcohol passes into the receiver flask. Note: this final distilled product can be filtered through charcoal by placing a layer of charcoal briquettes or granules into the bottom of the filter funnel (over the filter paper), and then filtering the ethyl alcohol there through several times. See the following illustration for details on the appropriate distillation apparatus to be used for the first distillation.

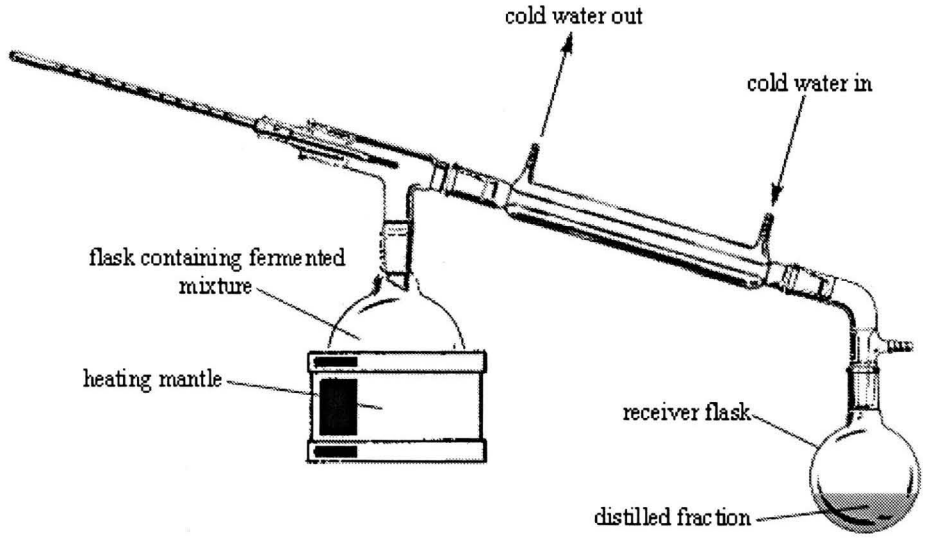
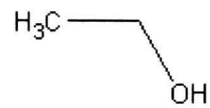


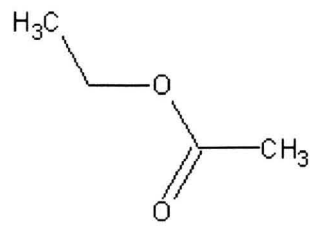
Figure 032. Apparatus for the distillation of ethyl alcohol (ethanol).

99% Ethyl alcohol



99% Ethyl alcohol is a colorless, very mobile and flammable liquid with a pleasant odor. Pure ethanol is tasteless. It rapidly absorbs water from the air, from which is forms an azeotrope of 95% ethyl alcohol. It is miscible with water, alcohol, ether, and many common organic solvents. 99% ethanol is toxic, and ingestion can cause poisoning. It is prepared by reacting ethylene gas with sulfuric acid, followed by distillation in the presence of minute amounts of water. It can also be made by double distillation of fermented cocktails, followed by treatment with metallic sodium to remove the water of azeotrope. 99% Ethyl alcohol can be obtained by salting out vodka (see 99% isopropyl alcohol), treating the recovered upper ethyl alcohol layer with large amounts of anhydrous magnesium sulfate, filtering, and then distilling the mixture to recover 99% ethyl alcohol.

Ethyl acetate

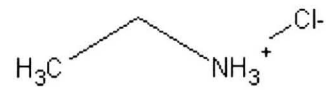


Ethyl acetate is a clear, volatile, and flammable liquid with a pleasant, fruity odor. It has a pleasant taste when diluted. Ethyl acetate slowly decomposes by moisture. It has a boiling point of 77 Celsius, and a melting point of -83 Celsius. Ethyl acetate is moderately soluble in water (1 milliliter in 10 milliliters of water), but is miscible with alcohol, acetone, chloroform, and ether. It forms a azeotropic mixture with water (6% by weight with a boiling point of 70 Celsius). Ethyl acetate can be prepared by distilling a mixture of ethanol and acetic acid in the presence of a few drops of 98% sulfuric acid. Ethyl acetate is a widely available commercial chemical.

40% Ethylamine solution

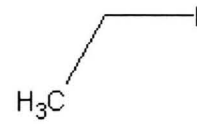
Prepare by dissolving 90 grams of ethylamine hydrochloride into 75 milliliters of ice-cold water, and then add in 58 grams of anhydrous sodium carbonate. Thereafter, stir the entire reaction mixture for about 1 hour at 0 Celsius, and then filter-off the sodium chloride.

Ethylamine hydrochloride



Ethylamine hydrochloride forms colorless to white crystals with a melting point of 110 Celsius. The crystals are readily soluble in water and alcohol, but not very soluble in most organic solvents. It is prepared by reacting ethyl iodide with excess liquid ammonia, followed by evaporation to remove excess ammonia, and then recrystallizing the left over residue from water.

Ethyl iodide



Ethyl iodide forms an oily colorless liquid with an ethyl like odor. It is colorless when freshly prepared, but turns yellow on standing, and turns red on prolonged exposure to light and air. The liquid has a melting point of –108 Celsius, and a boiling point of 72 Celsius. Ethyl iodide is insoluble in water, but readily soluble in alcohol, ether, and most organic solvents. It can be made by reacting a mixture of 95% ethyl alcohol and iodine, with red phosphorus, followed by fractional distillation to recover the ethyl iodide. Some ethyl iodide can be made by mixing 95% ethyl alcohol with potassium or sodium iodide and then adding in a 25% sulfuric acid solution. The resulting reaction mixture is then extracted with ether, and the ether extracts are then combined and then distilled. The resulting distilled mixture is then fractionally distilled to separate the ethyl iodide from the alcohol.

Ferric chloride (anhydrous)

$$FeCl_3$$

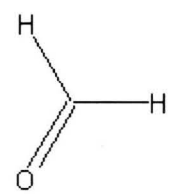
Ferric chloride forms dark brown to brownish-black crystals, which begin to volatize when heated to about 300 celsius. The crystals can be melted at 316 Celsius under slight pressure. The anhydrous material readily absorbs moisture from the air forming the hexahydrate. Ferric chloride is soluble in alcohol, acetone, and slightly soluble in carbon disulfide. It can be made by passing chlorine gas over heated scrap iron.

Ferric chloride hexahydrate

$$FeCl_3 \cdot 6H_2O$$

The hexahydrate forms yellowish-brown crystals or orange like powder. The crystals may have a slight odor of hydrogen chloride, with a melting point of about 37 Celsius. Ferric chloride hexahydrate is readily soluble in water and alcohol, acetone, and ether. It can be made by reacting chlorine with iron in the presence of water; for example, placing nails or pieces of iron in a glass of water, and then passing chlorine gas there into.

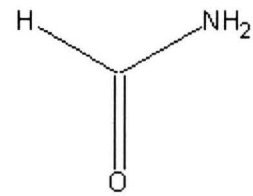
37% Formaldehyde



Pure formaldehyde is a colorless flammable gas with a pungent suffocating odor. The pure gas has a melting point of –92 Celsius, and a boiling point of –19 Celsius. In most cases, formaldehyde is handled as a 37% solution in water, from which methanol has been added to prevent polymerization. 37% formaldehyde solution is a colorless liquid with a pungent odor. Vapors from 37% formaldehyde can produce dizziness and headache upon inhalation, and maximum ventilation should be used when using. The colorless liquid solution may become cloudy on standing as a result of polymerization. 37% Formaldehyde slowly oxidizes to formic acid on standing. The liquid solution has a boiling point of 96 Celsius. It is miscible

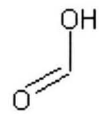
with water, alcohol, and acetone. Formaldehyde can be made by the oxidation of methanol with potassium dichromate, chromium trioxide, or other moderate oxidizing agents.

Formamide



Formamide forms a colorless oily syrupy liquid with a very viscous nature. The liquid has a melting point of 3 Celsius, and a boiling point of 211 Celsius. The liquid begins to slowly decompose into ammonia and carbon dioxide when heated to 180 Celsius. Formamide is readily soluble in most organic solvents, and it makes a good solvent for a great many materials. It can be made in the lab by reacting formyl chloride with ammonia under reflux conditions, then treating the cooled reaction mixture with sodium carbonate, followed by treating the alkaline mixture with water, extracting this aqueous mixture with ether, and then evaporation of the ether to leave behind the desired product.

Formic acid. *Methanoic acid*

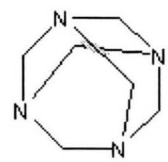


Formic acid is a colorless liquid with a pungent odor. It has a boiling point of 101 Celsius, and a melting point of 8.4 Celsius. Formic acid is a toxic liquid. It forms an azeotropic mixture with water containing 77.5% formic acid by weight with boiling point of 107 Celsius. Formic acid is a strong reducing agent, and is capable of reducing a variety of oxidizing agents. It is miscible with water, ether, acetone, ethyl acetate, and methanol. Formic acid is slightly soluble in benzene, and toluene. Small amounts of formic acid can be obtained by destructively distilling red ants. Formic acid is prepared on a large scale by the catalytic oxidation of methanol, and is widely available commercially. Formic acid can be made in the lab by oxidizing methanol with potassium permanganate, followed by distillation.

90% Formic acid solution: Prepare by dissolving 100 grams of 99% formic acid into 10 milliliters of water.

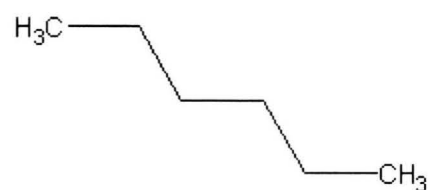
80% Formic acid (concentrated formic acid): Prepare by dissolving 100 grams of 99% formic acid into 25 milliliters of water. Note: can be purchased commercially.

Hexamine. *Methenamine; 1,3,5,7-tetraazatricyclo[3.3.1.-1.3,7]decane; hexamethylenetetramine*



Hexamine forms colorless, odorless, or white granules, powder, or crystals. It sublimes at 263 Celsius without melting, but is volatile below this temperature. Hexamine burns with a smokeless flame, and is used in solid camping fuel pellets (in high purity). 1 gram of hexamine dissolves in 1.5 milliliters water, and 12.5 milliliters of alcohol. It is insoluble in ether. Hexamine is a widely available, and cheap commercial chemical.

Hexane. Hexanes



Hexanes are a colorless, very volatile liquid with a faint, peculiar odor. It is rarely sold as n-hexane but usually admixed with hexane isomers simply called "hexanes", but marketed as "hexane". Hexane has a boiling point of 69 Celsius, and a melting point of –100 Celsius. It is insoluble in water, but miscible with alcohol, chloroform, and ether. Hexane is a major component of gasoline, and can be distilled from the gasoline using a multiple-path distillation apparatus. Hexane is obtained commercially from petroleum, and is a widely available commercial chemical.

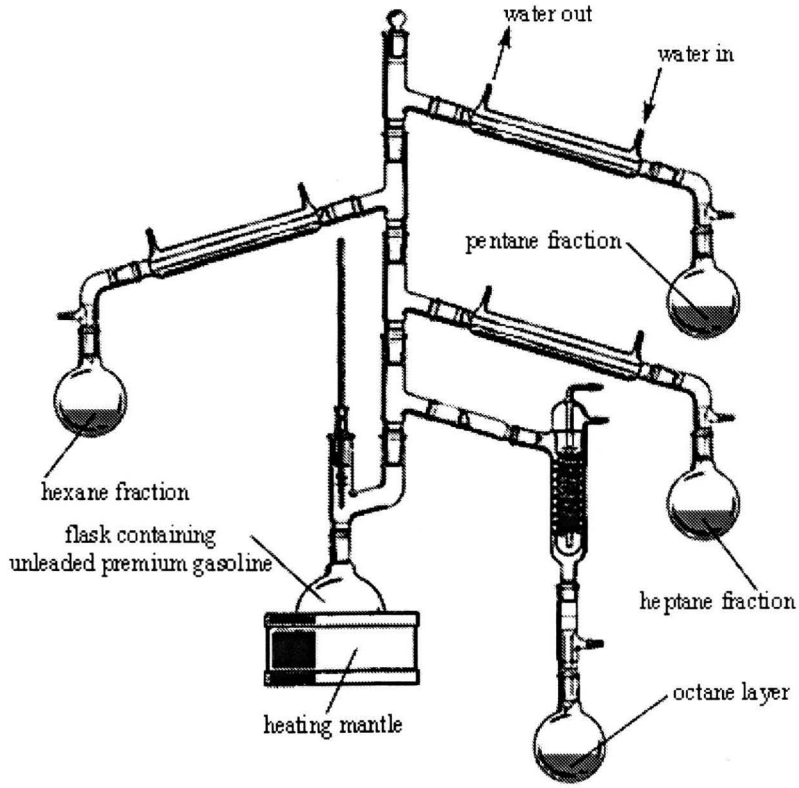


Figure 033. Apparatus for the distillation of unleaded premium gasoline (carefully distill at 70 Celsius).

47 to 48% Hydrobromic acid

Hydrobromic acid forms a colorless to slightly yellow liquid, which slowly darkens on exposure to light. The acid is miscible with water and alcohol. Hydrobromic acid forms a constant boiling mixture containing 47% hydrobromic acid by weight, with a boiling point of 126 Celsius. Hydrobromic acid is prepared by mixing dilute sulfuric acid (25%) with a concentrated solution of potassium or sodium bromide at 0 Celsius, and then distilling the entire mixture at 126 Celsius to obtain the 47% acid.

Hydrochloric acid. *35 - 38% Hydrochloric acid; Water solution of hydrogen chloride*

HCl

35 – 38% Hydrochloric acid is commonly referred to as concentrated hydrochloric acid. It is a highly corrosive liquid, which evolves choking and corrosive fumes. Some brands of concentrated hydrochloric acid may be colored yellow due to iron. Concentrated hydrochloric acid turns yellow when exposed to sunlight. It forms a constant boiling mixture with water containing 20% hydrogen chloride by weight, and boiling at 108 Celsius. Avoid contact with fumes, and keep in tightly sealed amber glass bottles. Concentrated hydrochloric is prepared on an industrial scale by condensing hydrogen chloride vapors, produced as a by-product in the production of chlorinated hydrocarbons. It can be prepared in the laboratory by mixing sodium chloride with 98% sulfuric acid and then passing the hydrogen chloride vapors into a quantitative amount of water. Concentrated hydrochloric acid is a widely available commercial chemical. It is available in most hardware stores sold under the name "Muriatic acid" (20 to 31% hydrogen chloride by weight). It can be made by dripping concentrated sulfuric acid onto sodium chloride, and then bubbling the resulting hydrogen chloride vapors into a minimal amount of ice water. It can also be made by bubbling moist chlorine gas (generated by electrolysis of sodium chloride brine) into hexane containing aluminum foil pieces, and then bubbling the liberated hydrogen chloride vapors into a minimal amount of ice water.

20% Hydrochloric acid solution: Prepare by diluting 140 grams of 35 to 38% hydrochloric acid into 110 milliliters of water.

15% Hydrochloric acid solution: Prepare by diluting 100 grams of 35 to 38% hydrochloric acid into 130 milliliters of water.

10% Hydrochloric acid solution: Prepare by dissolving 100 grams of 35 – 38% hydrochloric acid into 250 milliliters of water.

7% Hydrochloric acid solution: Prepare by diluting 100 grams of 35 to 38% hydrochloric acid into 400 milliliters of water.

5% Hydrochloric acid solution: Prepare by diluting 100 grams of 35 to 38% hydrochloric acid into 600 milliliters of water.

Hydrogen

H_2

Hydrogen is a colorless, tasteless, highly flammable and explosive gas. Hydrogen is a strong reducing agent when in the presence of a suitable catalyst such as platinum, palladium, nickel, and the like. Hydrogen gas is very difficult to handle and store, but can be stored in compressed cylinders; these cylinders are often hard to purchase and ship, but hydrogen can easily be prepared by reacting dilute hydrochloric acid with zinc, or aluminum, and collecting the resulting hydrogen gas. Note: iron should be avoided as it contains impurities leading to the formation of metal hydrides such as arsine and stibnine, which contaminate the hydrogen (gives the hydrogen a strange metallic like garlic odor). Hydrogen can also be obtained by the electrolysis of acidic or basic water solutions, or by treating a 25% sodium hydroxide solution with aluminum foil.

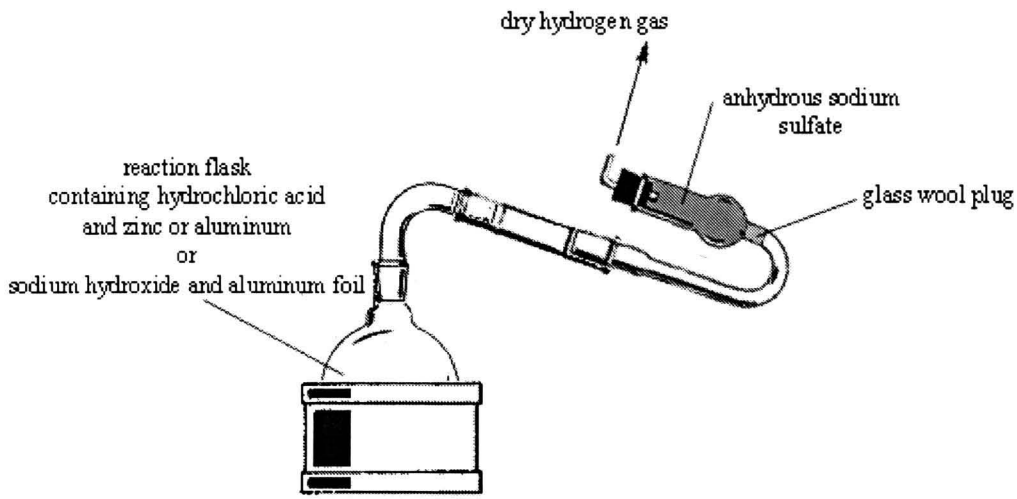


Figure 034. Apparatus for the generation of hydrogen gas. Heat can be applied to speed up the reaction.

Hydrogen bromide

HBr

Hydrogen bromide is a colorless and corrosive gas, which is non-flammable. The gas has a melting point of –87 Celsius, and a boiling point of –67 Celsius. It is readily soluble in water and alcohol. It can be made by slowly dripping moist liquid bromine or slowly adding moist bromine vapor into a dry hexane mixture containing aluminum foil pieces, and then bubbling the hydrogen bromide vapors into a minimal amount of ice water. Hydrogen bromide can also be made by brominating an organic compound in the presence of sunlight, and then bubbling the liberated hydrogen bromide by-product into a minimal amount of ice water.

Hydrogen chloride (anhydrous)

Anhydrous hydrogen chloride is a very corrosive, non-flammable gas, with a highly irritating vapor. Hydrogen chloride is very soluble in water, forming a fuming liquid; vide supra, hydrochloric acid. It has a melting point of –114 Celsius, and a boiling point of –85 Celsius. It is prepared by the reaction of concentrated sulfuric acid upon table salt, or by the action of chlorine upon organic compounds. The latter being the chief source of hydrochloric acid.

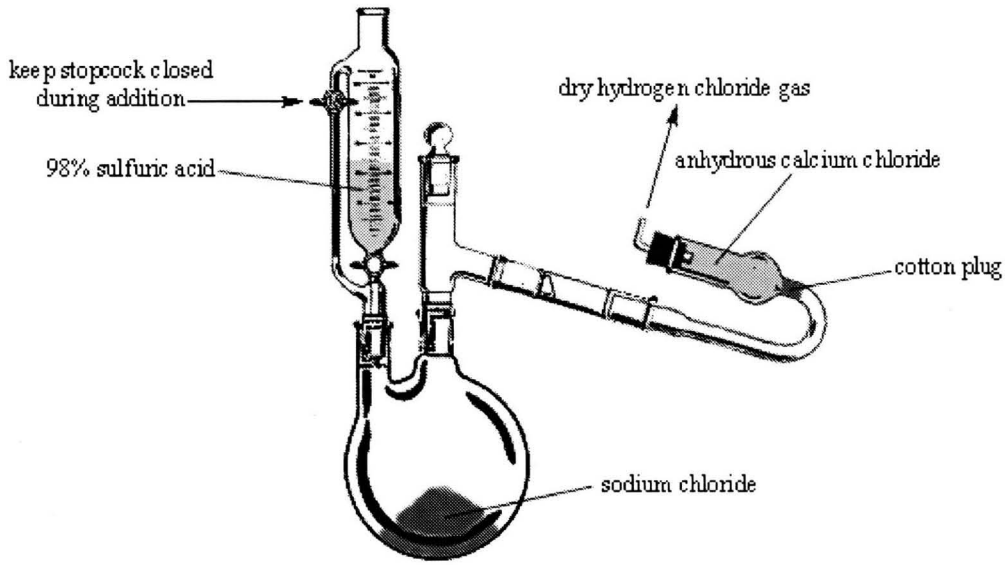


Figure 035. Apparatus for the preparation anhydrous hydrogen chloride

50% Hydrogen peroxide

$$H_2O_2$$

50% hydrogen peroxide forms a colorless oily syrupy liquid with a strange taste. It is a powerful oxidizer and should be kept away from combustible materials, and reducing agents, especially finely divided metals and dust. 50% Hydrogen peroxide is commercially available, and is the most common commercial concentration of the peroxide sold. Most commercial brands come stabilized, but still must be protected from dust, heat, and light.

35% Hydrogen peroxide solution: Prepare by diluting 100 grams of 50% hydrogen peroxide into 43 milliliters of water.

30% Hydrogen peroxide

$$H_2O_2$$

30% Hydrogen peroxide is a colorless syrupy liquid. The liquid is a strong oxidizer and solutions should be stored in cool dry places away from combustible materials and reducing agents. 30% Hydrogen peroxide should be handled with care, and gloves should be worn when handling. Avoid contact with all materials, including dust. 30% Hydrogen peroxide is available from various sources.

6% Hydrogen peroxide solution: Prepare by diluting 165 grams of 30% hydrogen peroxide into 705 milliliters of water.

Hydrogen sulfide gas

$$H_2S$$

Hydrogen sulfide is a flammable, poisonous gas with disagreeable odor of rotten eggs. It can be detected by the human nose in extremely small quantities. Hydrogen sulfide has a sweetish taste. Hydrogen sulfide burns in air with a pale blue flame. It has a melting point of –85 Celsius, and a boiling point of –60 Celsius. Hydrogen sulfide is insoluble in water, and not very soluble in alcohol. It is soluble in glycerol, gasoline, kerosene, carbon disulfide, and crude oil. Hydrogen sulfide is a highly toxic gas, and inhalation can be fatal. Use maximum ventilation when handling. It is evolved from many different natural environmental sources including bacterial decomposition of vegetable and animal proteins, natural springs, natural gas deposits, and volcanoes. Hydrogen sulfide can be obtained from the distillation of petroleum. Hydrogen sulfide is prepared in the laboratory by dropping an acid (usually sulfuric or hydrochloric) onto a metal sulfide such as sodium sulfide, or calcium sulfide (calcium sulfide is prepared by roasting calcium sulfate with charcoal at 1000 Celsius). Hydrogen sulfide is commercially available, but shipping regulations may decrease sale.

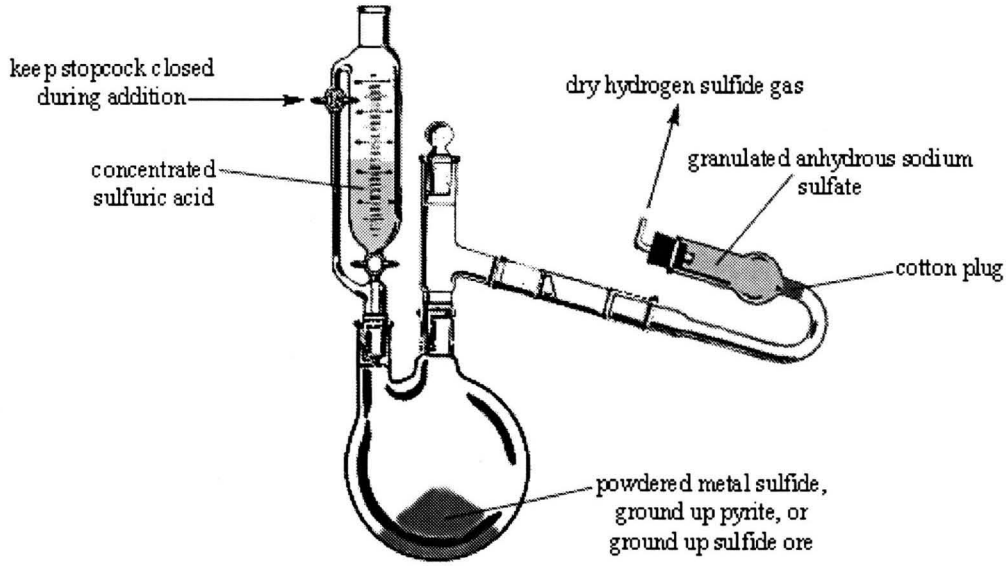


Figure 036. Apparatus for the preparation of hydrogen sulfide gas.

Hydroiodic acid

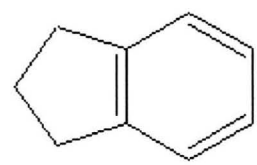
HI

Hydroiodic acid is a colorless to yellowish to brownish liquid. It is colorless when freshly prepared, but rapidly turns yellow due to oxidation. Hydroiodic acid should be used within 2 weeks of its preparation, and it will turn brown on standing due to oxidation; however, these brownish solutions can be regenerated by adding in hypophosphorus acid. Hydroiodic acid forms an azeotrope with water containing 57% acid by weight with a constant boiling temperature of 127 Celsius. Hydroiodic acid is readily prepared by bubbling hydrogen sulfide gas into a suspension of iodine in water, and then filtering-off the precipitated sulfur.

Method of preparing 46% hydroiodic acid

Into a suitable reaction flask (quipped with motorized stirrer or other stirring means, and gas inlet tube, place 32 grams of iodine crystals, followed by 36 milliliters of water. Then place this reaction flask into an ice bath, and chill to about 0 Celsius. Thereafter, bubble into this iodine mixture, 5 grams of hydrogen sulfide gas. During the addition of the hydrogen sulfide gas, rapidly stir the iodine mixture and maintain its temperature at 0 Celsius. After the addition of the hydrogen sulfide gas, continue to rapidly stir the entire reaction mixture for about 30 minutes at 0 Celsius, and then filter-off the precipitated sulfur. Now, add to this filtered reaction mixture, 5 grams of iodine crystals, and then store this reaction mixture (which will be the 47% hydroiodic acid) in a refrigerator at 5 Celsius until use.

Indan



Indan forms a liquid with a melting point of –51 Celsius and a boiling point of 180 Celsius. The liquid is insoluble in water, but soluble in the usual organic solvents. Indan is available from commercial sources, but may be somewhat expensive.

Iodine

I_2

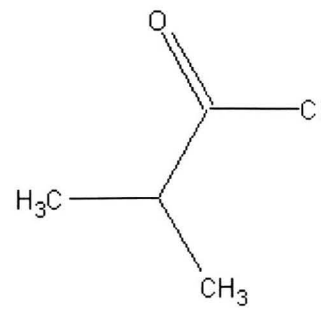
Iodine forms blackish to purplish-black plates, powder, or granules with a characteristic metallic luster and peculiar odor. It forms a purple vapor when gently heated, and readily volatizes. It has a melting point of 114 Celsius and boiling point of 185 Celsius. Iodine is soluble in aqueous solutions of potassium or sodium iodide, and is soluble benzene, ethyl alcohol, ether, cyclohexane, and methylene chloride. Iodine is capable of producing eye, nose, and throat irritation so wear gloves when handling and use ventilation. Iodine is readily obtainable by bubbling chlorine gas into a solution of potassium or sodium iodide at room temperature, then filtering-off the precipitated iodine, followed by sublimation to purify the iodine. Iodine can also be obtained by treating solutions of potassium or sodium iodide with bleaching powder, potassium permanganate solutions, or with excess bleach (Clorox), then filtering-off the precipitated iodine crystals, followed by sublimation to purify the iodine.

Iron

Fe

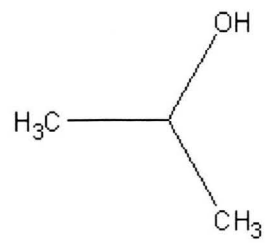
Iron is a silver-white to gray metal, which is ductile and malleable, and capable of being magnetic. Iron is by far one of the most common metals known in the world, and is readily available in about every shape, size, and form imaginable. Iron is relatively stable in air, when admixed with stabilizers such as in steel, but pure iron (cast iron) is relatively reactive and readily oxidizes in air forming rust. Powdered iron is black to blackish gray, and is capable of igniting readily in the presence of any source of ignition, even when touched with a 9-volt battery. Iron powder is a strong reducing agent, and is also readily available from a number of sources. Iron fillings can be obtained by electrolyzing a solution of ferrous chloride or dilute hydrochloric acid with an iron anode, and a graphite cathode. The iron fillings, chips, or flakes collect on the graphite electrode.

Isobutryl chloride



Isobutryl chloride forms a colorless to slightly yellow liquid with a odor similar to acetyl chloride. It reacts with water and alcohol, and should be stored in tightly sealed bottles. It can be made by reacting isobutyric acid with sulfur chloride, thionly chloride, sulfuryl chloride, or phosphorus trichloride.

99% Isopropyl alcohol



Isopropyl alcohol is a colorless flammable liquid with a characteristic acetone/ethanol odor. It has a melting point of –88 Celsius, and a boiling point of 82 Celsius. Isopropyl alcohol is miscible in water, alcohol, ether, and chloroform. It is insoluble in salt solutions, and can be extracted from water solutions by the addition of excess salt. Isopropyl alcohol is very common, and is sold under the name "rubbing alcohol". It is manufactured on a large scale by reacting propylene gas with sulfuric acid followed by treatment with water, and subsequent distillation. To obtain 99% isopropyl alcohol from rubbing alcohol, place 250 milliliters or more of your rubbing alcohol into a seperatory funnel, and then add in about 50 to 90 grams of pickling salt. Then shake the entire mixture vigorously for about 10 minutes. Thereafter, allow the seperatory funnel to stand for about 10 minutes, and then drain-off the bottom water layer. Note: excess salt will drain out with the water layer. Once the water layer has been removed, place the upper isopropyl alcohol layer into beaker or flask, and then add in 50 grams of anhydrous

magnesium sulfate. Then stir the entire mixture for about 30 minutes (to absorb moisture). Thereafter, filter-off the magnesium sulfate, and then place the dried filtered isopropyl alcohol into a distillation apparatus, and distill the alcohol at 82 Celsius. When most of the alcohol has distilled over, stop the distillation process and then recover the isopropyl alcohol from the receiver flask. This distilled alcohol will be about 95 to 99% pure isopropyl alcohol, well suitable for use in processes where 99% isopropyl alcohol is needed.

Lead oxide

$$PbO$$

Lead oxide exists in two forms, the first form being a reddish yellow powder and the other form, a yellow powder. Each form is stable when heated up to 489 Celsius. The powder slowly oxidizes on exposure to air, so it should be stored in airtight containers. The compound is insoluble in water and alcohol, and the usual organic solvents. Lead oxide is toxic, and ingestion and inhalation of the dust should be avoided. It can be made by dissolving lead into hot dilute acetic acid, followed by recrystallization of the acetate salt there from, then precipitating the carbonate by treating an aqueous solution of the acetate salt with an aqueous solution of sodium carbonate, filtering-off the carbonate, and then roasting the carbonate at high temperature to form the oxide.

Lithium aluminum hydride

$$LiAlH_4$$

Lithium aluminum hydride forms white to grayish crystals. The compound decomposes when heated to above 125 Celsius, and it slowly looses hydrogen gas when heated to 120 Celsius. Lithium aluminum hydride is very reactive and decomposes in the presence of moisture, water, and a great many compounds. It should be stored in tightly sealed bottles in a cool dry place. It is made by reacting excess lithium hydride with an ether solution of aluminum chloride at 0 Celsius, followed by evaporation of the ether.

Magnesium

$$Mg$$

Magnesium is a slivery-white metal with a melting point of 651 Celsius, and a boiling point of 1100 Celsius. The metal is flammable, and burns with an intense light; even large bars are combustible but require much heat for ignition. Powdered magnesium burns violently and rapidly. The metal is insoluble in water, and only slowly reacts with water forming magnesium hydroxide. The metal readily reacts with dilute acids forming the corresponding salts, and it reacts with ammonium salt solutions forming double salts. The metal is capable of reducing many gases including carbon dioxide, sulfur dioxide, nitric oxide, and nitrous oxide when heated. Magnesium is readily available and can be purchased in many forms and sizes. It is prepared on an industrial scale by the electrolytic reduction of molten magnesium chloride.

Magnesium sulfate

$$MgSO_4$$

The monohydrate occurs in nature as the mineral kieserite. The monohydrate can be converted to the anhydrous form by heating to 250 Celsius for 1 hour. The monhydrate can be prepared by heating the heptahydrate (Epsom salt) to 120 Celsius for 1 hour. The trihydrate forms odorless, coloress crystals. The trihydrtae is converted into the anhydrous salt by heating to 250 Celsius for 1 hour. The trihydrate can be prepared by heating the heptahydrate to 80 Celsius for 1 hour. The heptahydrate occurs in the nature as the mineral epsomite. It forms efflorescent crystals, or a white powder with a bitter, saline, and cool taste. The heptahydrate is soluble in water, but insoluble in alcohol. The heptahydrate is converted to the anhydrous salt by heating to 250 Celsius for 1 hour. The anhydrous salt is highly hygroscopic and absorbs moisture rapidly. The anhydrous salt is a common drying agent, but cannot be used to dry ammonia.

Manganese dioxide

$$MnO_2$$

Manganese dioxide forms a black powder. The compound is a strong oxidizer and it should be kept away from combustible materials and reducing agents. It is made by reducing potassium permanganate, and is a major by-product in oxidations involving potassium permanganate. The compound exists naturally, but is readily available from commercial sources.

Mercury-II-chloride. *Mercuric chloride*

$$HgCl_2$$

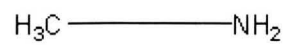
Mercury chloride forms white granules or powder with a melting point of 277 Celsius. The salt volatizes at 300 Celsius but can volatize at lower temperatures. Mercury chloride is a violent poison, and ingestion, inhalation, and skin absorption should be avoided at all costs. The salt is only slightly soluble in water, alcohol, acetic acid, glycerol, and ether. The salt is moderately soluble in methanol, acetone, and ethyl acetate. It can be prepared by refluxing concentrated hydrochloric acid with mercury-II-oxide.

Methyl alcohol. Methanol

Methanol is a flammable, poisonous, and mobile liquid. It has a slight alcoholic odor when pure, and burns with a non-luminous, bluish flame (flames from burning methanol are difficult to see). Methanol has a melting point of –97.8 Celsius, and a boiling point of 65 Celsius. It is miscible with water, alcohol, ether, benzene, acetone, and most organic solvents. Methanol forms azeotropes with many solvents, and it dissolves many inorganic substances. Methanol is a toxic liquid, and ingestion leads to headache, vision problems, and death. The average fatal dose is usually 100 to 200 milliliters, but death has occurred from as little as 30 milliliters. Methanol can be prepared by destructive distillation of wood (heating wood to a high temperature in the absence of air), and then condensing the vapors. The condensed vapors are then distilled to separate the methanol from the acetic acid. Methanol is prepared industrial from carbon monoxide and carbon dioxide. Methanol is a readily available commercial chemical and is the chief ingredient in windshield wiper fluid; from which it can be separated by distillation.

70% Methyl alcohol solution: Prepare by diluting 100 grams of 99% methyl alcohol into 42 milliliters of water.

Methylamine

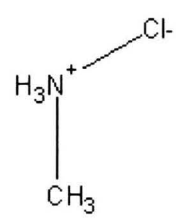


Methylamine forms a flammable colorless gas. It is soluble in water, and is usually sold as a 33% aqueous solution. The gas is also soluble in alcohol and toluene. Methylamine is insoluble in chloroform, acetone, most ethers, and ethyl acetate. Methylamine is widely sold as the hydrochloride, from which it forms colorless to white crystals. Methylamine is readily obtained by heating 37% formaldehyde with ammonium chloride, and then treating the resulting mixture with sodium carbonate, followed by filtration.

40% Methylamine solution

40% Methylamine solution can be prepared by dissolving 110 grams of methylamine hydrochloride into 75 milliliters of ice-cold water. Thereafter, add in 86 grams of anhydrous sodium carbonate, and then stir the entire mixture for about 1 hour at 0 Celsius. Thereafter, filter-off the insoluble sodium chloride.

Methylamine hydrochloride



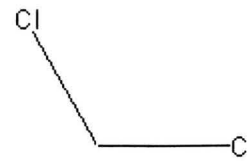
Methylamine hydrochloride forms deliquescent crystals with a melting point of 228 Celsius, whereupon it begins to sublime. The crystals are readily soluble in water and in alcohol, but insoluble in most organic solvents. It is prepared by heating 37% formaldehyde with ammonium chloride, and then recrystallizing the methylamine hydrochloride from the reaction mixture.

50% Methylamine hydrochloride solution: Prepare by dissolving 100 grams of methylamine hydrochloride into 100 grams of warm water.

Methylene bromide

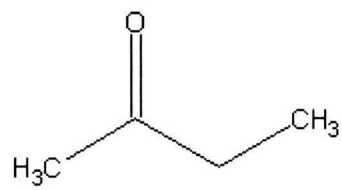
Methylene bromide forms a colorless liquid with a melting point of –53 Celsius, and a boiling point of 97 Celsius. The liquid is insoluble in water, but miscible with alcohol, ether, and acetone. It can be manufactured along with bromochloromethane by treating methylene chloride with a reactive brominated compound such as phosphors tribromide or sulfur bromide in the presence of a catalyst.

Methylene chloride. *Dichloromethane*



Methylene chloride is a colorless, highly volatile liquid, which is insoluble in water, but miscible with ether, and alcohol. It has a boiling point of 40 Celsius, and a melting point of -95 Celsius. Methylene chloride is a powerful, and widely used solvent that is readily available commercially. Methylene chloride is obtained by condensing the vapors obtained by the reaction of dry chlorine with dry methane. The methylene chloride is then separated from the chloroform and carbon tetrachloride by distillation.

Methyl ethyl ketone. Ethyl methyl ketone

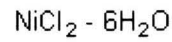


Methyl ethyl ketone is a colorless volatile liquid with a melting point of –86 Celsius, and a boiling point of 80 Celsius. Methyl ethyl ketone is slightly soluble in water, but miscible with alcohol, ether, and benzene. It forms an azeotrope with water containing 89% methyl ethyl ketone by weight. Methyl ethyl ketone is similar to acetone and has a related odor. It is made by the oxidation of 2-butanol.

Methyl iodide

Methyl iodide forms a colorless very heavy, and highly refractive liquid, which darkens on exposure to light, air, and moisture. The liquid has a melting point of 6 Celsius, and a boiling point of 181 Celsius. Methyl iodide tends to solidify when cooled to 5 Celsius, from where it forms colorless leaflets. Methyl iodide can be prepared in the lab by heating iodoform with sodium acetate in the presence of alcohol. Iodoform is prepared by reacting iodine with sodium hydroxide solution and then adding in acetone (similar to the preparation of chloroform).

Nickel chloride hexahydrate



Nickel chloride hexahydrate forms greenish crystals, powder, or granules with a slight odor of hydrogen chloride. The crystals are soluble in water and alcohol. The crystals can be made by dissolving nickel metal into dilute hydrochloric acid, and then recrystallizing the salt from the reaction mixture.

99%+ Anhydrous nitric acid (absolute nitric acid)

99% nitric acid is a colorless (when freshly prepared), highly fuming, and poisonous liquid. It turns yellow to dark-red on standing, and has a melting point of –42 Celsius. 99% nitric acid is a powerful oxidizer. It reacts violently with many substances. 99% nitric acid should be used right after preparation. 99% Nitric acid is commercially available, but shipping regulations restrict its shipment to most locations. *Warning! 99% Nitric acid is very poisonous, and corrosive liquid which evolves large amounts of poisonous fumes. Wear gloves and proper laboratory clothing (lab coat; boots, face shield) when handling this substance and use maximum ventilation.*

Hazards: Carryout the distillation with extreme caution. Do not heat the methylene chloride/nitric acid mixture above 40 Celsius, and use proper ventilation. Nitrogen oxide gases will develop so be prepared. Carryout the distillation away from direct sun light.

Into an appropriate sized beaker place 47 milliliters of 98% sulfuric, and then 53 milliliters of 70% nitric acid. Then extract this acid mixture with seven 100-millilter portions of methylene chloride. Afterwards, combine all seven portions of methylene chloride (if not already done so). The result will be a 99% nitric acid solution in methylene chloride. This methylene chloride/nitric acid mixture can be used directly in nitrations (if desired), or separated to recover pure 99% nitric acid. To separate the mixture, place the mixture into a distillation apparatus, and then carefully distill at 40 Celsius until no more methylene chloride is collected in the receiver flask. *Note: Take caution when distilling and use proper ventilation because decomposition of the nitric acid might result forming reddish-brown fumes of nitrogen oxides. If the nitric acid begins to decompose during the distillation, don't worry and continue the distillation. After all the methylene chloride has been removed, the result might be a reddish-brown highly fuming liquid. This reddish-brown fuming liquid is suitable for use as 99% nitric acid.*

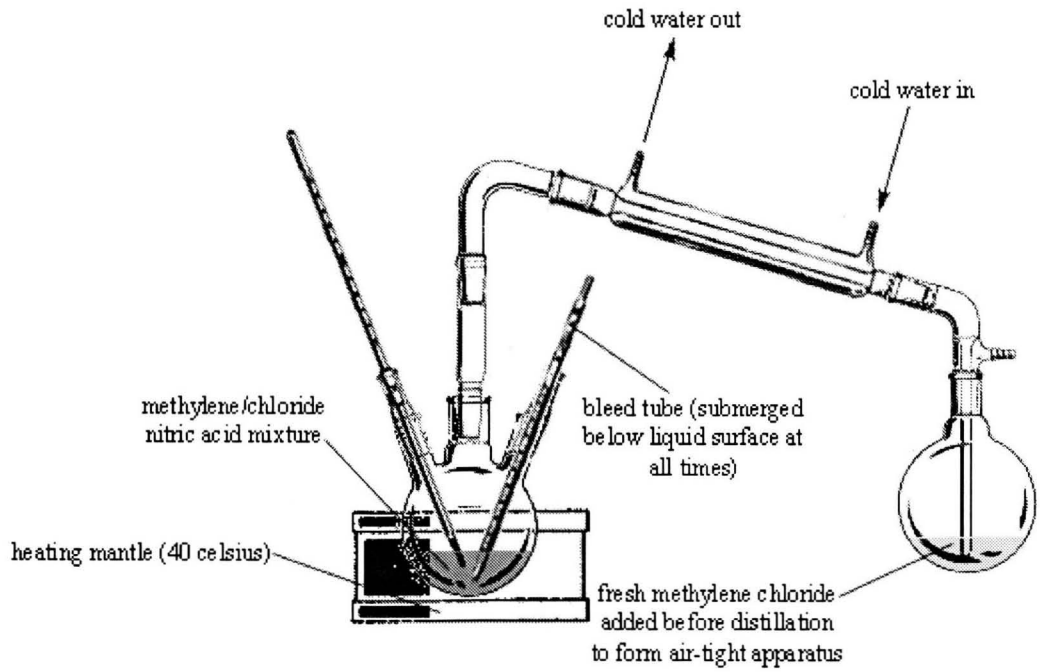


Figure 037. Apparatus for the distillation of methylene chloride to collect pure nitric acid

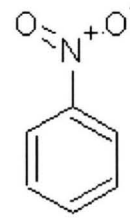
Note: Any concentration of nitric acid can be used instead of just 70% nitric acid. When using nitric acid concentrations below 70% by weight, simply mix the dilute nitric acid with excess sulfuric acid. For example, mix 57 grams of 98% sulfuric acid with 53 milliliters of 60% nitric acid, mix 67grams of 98% sulfuric acid with 53 milliliters of 50% nitric acid, mix 77 grams of 98% sulfuric acid with 53 milliliters of 40% nitric acid, mix 87 grams of sulfuric acid with 53 milliliters of 30% nitric acid, mix 97 grams of 98% sulfuric acid with 53 milliliters of 20% nitric acid, or mix 107 grams of 98% sulfuric acid with 53 milliliters of 10% nitric acid. After one of these mixings, extraction with methylene chloride with seven 100-milliliter portions is the next step. The result is the same as in the above procedure. To isolate the 99% nitric acid, follow the directions in the above procedure.

Alternative method for preparing 99% nitric acid

Hazards: Take caution when distilling and use proper ventilation because decomposition of the nitric acid might take place forming reddish-brown fumes of nitrogen oxides. If the nitric acid begins to decompose during the distillation, don't worry and continue the distillation. After all the methylene chloride has been removed, the result might be a reddish-brown highly fuming liquid. This reddish-brown fuming liquid is suitable for use as 99% nitric acid. Carryout the distillation with extreme caution. Do not heat the methylene chloride/nitric acid mixture above 40 Celsius, and use proper ventilation. Nitrogen oxide gases will develop so be prepared. Carryout the distillation away from direct sun light.

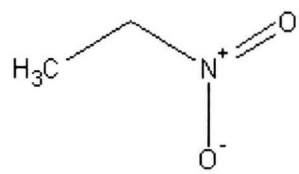
Place 194 grams of 98% sulfuric acid into a beaker, and then place the beaker in a ice bath and cool to 0 Celsius by means of an ice bath. When the sulfuric acid reaches a temperature of 0 Celsius, slowly add in portions, 100 grams of potassium nitrate or 84 grams of sodium nitrate over a period of 1 hour while stirring the 98% sulfuric acid and maintaining its temperature at 0 Celsius. After the addition of the potassium or sodium nitrate, slowly add over a period of one hour, 260 milliliters of cold water while continuously stirring the 98% sulfuric acid mixture and maintaining its temperature at 0 Celsius. Afterwards, remove the ice bath and then extract the acid mixture with seven 150-milliliter portions of methylene chloride. Then combine all seven portions of methylene chloride (if not already done so), and then place the methylene chloride into a distillation apparatus and carefully distill at 40 Celsius until no more methylene chloride is collected in the receiver flask.

Nitrobenzene



Nitrobenzene forms a colorless to pale yellow oily liquid with a melting point of 6 Celsius, and a boiling point of 211 Celsius. The liquid is toxic, and skin contact, ingestion, and inhalation should be avoided. Nitrobenzene is volatile with steam. It is insoluble in water, but soluble in alcohol, toluene, ether, and many oils. It can be made by reacting benzene with a mixture of nitric and sulfuric acids at 0 Celsius, followed by drowning the entire reaction mixture into ice water, recovering the upper oily layer, drying the upper oily layer with anhydrous sodium sulfate, and then filtering.

Nitroethane



Nitroethane forms a colorless to oily liquid with a pleasant odor. It has a melting point of –50 Celsius, and a boiling point of 115 Celsius. Nitroethane is only very slightly soluble in water, but miscible in alcohol, and ether. It is also soluble in chloroform. Nitroethane forms explosive salts when treated with strong bases. It can be made by reacting ethyl bromide with sodium nitrite.

Process for making nitroethane

Step 1: Preparation of ethyl bromide

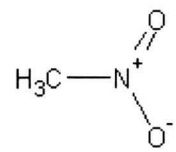
Into a standard flask, equipped with motorized stirrer or other stirring means, addition funnel, and thermometer, place 45 milliliters of ice water. Thereafter, slowly and carefully add in 75 milliliters of 98% sulfuric acid. Then place this acid mixture into an ice bath, and chill to about 0 Celsius. Afterwards, place 75 milliliters of 95% ethyl alcohol into the addition funnel, and then slowly add this ethyl alcohol, drop-wise, to the acid mixture. During the addition of the alcohol, stir the acid mixture and maintain its temperature around 0 Celsius at all times. After adding in the ethyl alcohol, slowly add in, 60 grams of potassium bromide or 52 grams of sodium bromide, in small portions at a time, over a period sufficient to keep the reaction mixture at 0 Celsius. After the addition of the bromide salt, continue to stir the entire reaction mixture for about 30 minutes, and thereafter, pour this entire reaction mixture into a distillation apparatus, and distill-off the ethyl bromide at 38 Celsius. When no more ethyl bromide passes over or is collected, stop the distillation process, and then recover the ethyl bromide from the receiver flask. Then add to this collected ethyl bromide, 10 grams of anhydrous calcium chloride, and then stir the entire mixture for

about 10 minutes—thereafter, filter-off the calcium chloride. Finally, re-distil this ethyl bromide using a fractional distillation apparatus at 38 Celsius. After the distillation process, collect the ethyl bromide and store it in an amber glass bottle in a refrigerator until use.

Step 2: Preparation of nitroethane

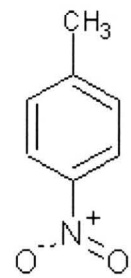
Into a standard flask, equipped with motorized stirrer or other stirring means, place 500 milliliters of dimethylformamide (DMF), followed by 30 grams of sodium nitrite. Thereafter, stir this entire mixture to form a uniform mix, and then place this mixture into a cold-water bath. Afterwards, carefully and gradually add in, 27 grams of ethyl bromide (prepared in step 1), over a period sufficient to keep the reaction mixtures temperature below 25 Celsius at all times. During the addition, rapidly stir the reaction mixture and maintain its temperature below 25 Celsius. After the addition of the ethyl bromide, continue to rapidly stir the reaction mixture for about 6 hours at a temperature below 30 Celsius. After 6 hours, pour the entire reaction mixture into a suitable sized beaker, and then add in 1250 milliliters of ice water. Thereafter, extract this aqueous mixture with five 90-milliliter portions of diethyl ether, and after the extraction process, combine all ether portions, if not already done so, and then wash this combined ether portion with three 75-milliliter portions of ice cold water. Note: after each extraction and washing portion, the ether will be the upper layer each time. After the washing portions, dry the collected washed ether portion by adding to it, 15 grams of anhydrous magnesium sulfate, and then stir the entire mixture for about 10 minutes—thereafter, filter-off the magnesium sulfate. Now, place this dried filtered ether mixture into a distillation apparatus, and distill-off the ether at 40 Celsius. When no more ether passes over or is collected, stop the distillation process, and then recover the left over remaining residue (after it has cooled). Finally, place this left over recovered residue into a distillation apparatus, and distill over the nitroethane at 115 Celsius. When no more nitroethane passes over or is collected, stop the distillation process, and recover the nitroethane (after it has cooled). Then place this nitroethane into an amber glass bottle and store it in a cool dry place until use.

Nitromethane



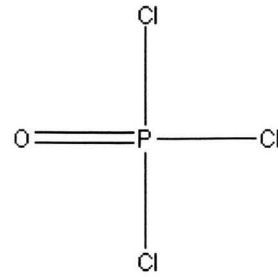
Nitromethane is an oily liquid with a strong odor. It is highly flammable, and has a melting point of -29 Celsius with a boiling point of 101 Celsius. Nitromethane is not very soluble in water, but is soluble in alcohol, ether, and DMF. It can form explosive salts with sodium, which ignite in contact with water. Nitromethane is used in liquid rocket fuels, and is produced on an industrial scale from vapor-phase oxidation of propane with nitric acid vapor. Nitromethane can be prepared on a laboratory scale by mixing sodium nitrite with sodium chloroacetate. Nitromethane is a widely available commercial chemical.

para-Nitro toluene



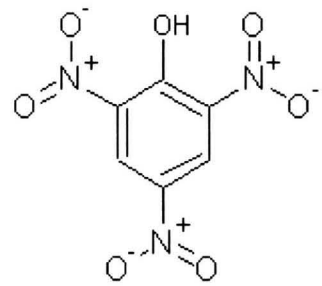
para-Nitro toluene forms a yellowish to reddish oily liquid, which is toxic by skin contact, ingestion, and inhalation. The compound can be obtained (along with small amounts of the ortho and meta isomers) by treating a mixture of nitric acid and sulfuric acid with toluene at 0 Celsius, and then drowning the entire reaction mixture into ice water after the reaction. The upper oily layer is then collected, dried with anhydrous sodium sulfate, filtered, and then stored until use.

Phosphorus oxychloride



Phosphorus oxychloride forms a colorless to slightly colored highly fuming liquid with a boiling point of 105 Celsius. It has a melting point of 1 Celsius, and solidifies when cooled to 0 Celsius. The liquid has a strong and irritating odor, and inhalation and skin contact of the fumes should be avoided. It decomposes vigorously in the presence of water or alcohol. Phosphorus oxychloride is a strong chlorinating agent and is used to chlorinate organic compounds. It can be prepared by the oxidation of phosphorus trichloride with potassium permanganate, bleaching powder, potassium dichromate, or other strong oxidizers—purification is accomplished by fractional distillation.

Picric acid



Picric acid forms yellow to pale yellow crystals with a melting point of 123 Celsius. The crystals explode when heated to 300 Celsius. Picric acid is insoluble in water, slightly soluble in alcohol and toluene, and relatively insoluble in methylene chloride and ether. Picric acid should be stored wet with 10% water, and it should be kept away from bases such as hydroxides and carbonates—as it forms shock sensitive compounds. Picric acid can be prepared by nitrating phenol with a mixture of nitric and sulfuric acids.

Potassium bisulfite

$KHSO_3$

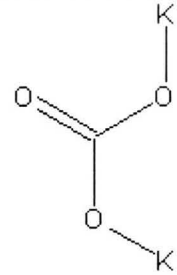
Potassium bisulfite forms white crystals, powder or granules with a peculiar odor. The crystals are soluble in water, but to a lesser extent then the sodium salt, and they are relatively insoluble in most organic solvents. Potassium bisulfite can be prepared by bubbling sulfur dioxide gas into a concentrated solution of potassium hydroxide or carbonate, and then filtering-off the precipitated bisulfite salt.

Potassium bromide

KBr

Potassium bromide forms colorless to white crystals or powder with a melting point of 730 Celsius. The crystals are soluble in water, but insoluble in alcohol and most other solvents. It is commercially available, but can be made by neutralizing potassium carbonate or hydroxide with bromine, followed by recrystallization.

Potassium carbonate



Potassium carbonate forms hygroscopic, odorless granules or white powder with a melting point of 891 Celsius. It is very soluble in water, but insoluble in alcohol. The water solution is strongly alkaline. The anhydrous salt forms the sesquihydrate when recrystallized from water solutions. The sesquihydrate forms small granular crystals. Potassium carbonate is simply made by roasting the bicarbonate for several hours. The bicarbonate is made by bubbling carbon dioxide gas or by placing dry ice into a concentrated solution of potassium hydroxide, followed by filtration. Potassium carbonate can also be made by roasting potassium chloride with magnesium carbonate, and then leaching out the magnesium chloride with 99% isopropyl alcohol, followed by filtering to remove the insoluble potassium carbonate, which is then briefly roasted to convert any hydrate to the anhydrous form.

30% Potassium carbonate solution: Prepare by adding and dissolving 100 grams of anhydrous potassium carbonate into 230 milliliters of water.

Potassium cyanide

KCN

Potassium cyanide forms white deliquescent powder or granules, or fused pieces with a slight odor of hydrogen cyanide. Potassium cyanide is slowly decomposed by air and carbon dioxide on standing, and should be kept in tightly sealed bottles. Potassium cyanide is a deadly poison, and ingestion of as little as 50 milligrams may be fetal. The salt has a melting point of 634 Celsius, and it is moderately soluble in water, glycerol, and only very slightly soluble in alcohol. Aqueous solutions of potassium cyanide are strongly alkaline, and rapidly decompose on standing. Potassium cyanide should be kept away from acids, and strong oxidizers. Avoid ingestion and skin absorption. It can be made by bubbling hydrogen cyanide (generated from potassium ferrocyanide and dilute sulfuric acid), into potassium hydroxide or carbonate, followed by recrystallization.

Potassium dichromate

$K_2Cr_2O_7$

Potassium dichromate forms brilliant orange crystals. The crystals are not hydroscopic or deliquescent in anyway, and can be stored for many years. Potassium dichromate is soluble in water, but insoluble in alcohol and most organic solvents. Aqueous solutions have a characteristic orange color. Potassium dichromate is a strong oxidizer and should be kept away from combustible materials and reducing agents. Potassium dichromate is readily available from a number of sources—photography supply companies.

Potassium fluoride

KF

Potassium fluoride forms white to colorless cubic crystals with a melting point of 860 Celsius. Potassium fluoride is toxic, and skin contact and ingestion should be avoided. The crystals are soluble in water, but insoluble in alcohol and most solvents. Potassium fluoride absorbs moisture from the air forming a dihydrate and a tetrahydrate. Aqueous solutions of potassium fluoride corrode glass, and should be stored in aluminum or plastic containers. It can be made by neutralizing hydrofluoric acid with potassium carbonate, followed by recrystallization of the potassium fluoride, and then followed by heating the hydrates of the potassium fluoride to form the anhydrous salt.

Potassium hydroxide

KOH

Potassium hydroxide forms white or slightly yellow lumps, rods, or pellets, which rapidly absorb moisture and carbon dioxide from the air. It has a melting point of 360 Celsius. Pure potassium hydroxide has a melting point of 380 Celsius. It is very soluble in water, alcohol, and glycerol generating much heat when dissolved. Potassium hydroxide, and its solutions are very corrosive to the skin, and can produce burns. Keep dry potassium hydroxide or its solutions in tightly sealed bottles. Potassium hydroxide is toxic and ingestion causes tissue damage. It is prepared by the same manner as sodium hydroxide, and is a widely available commercial chemical.

33% Potassium hydroxide solution: Prepare by adding and dissolving 100 grams of potassium hydroxide into 200 milliliters of ice water. Note: allow the solution to cool before using.

10% Potassium hydroxide in 95% ethyl alcohol: Prepare by adding and dissolving 50 grams of potassium hydroxide into 450 grams of 95% ethyl alcohol.

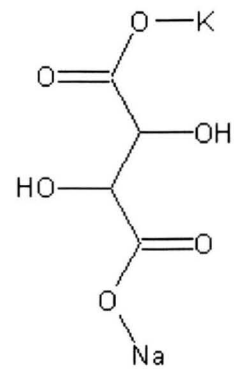
5% Potassium hydroxide solution: Prepare by dissolving 50 grams of potassium hydroxide into 950 milliliters of cold water.

Potassium permanganate

$$KMnO_4$$

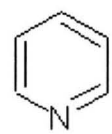
Potassium permanganate forms dark purple or bronze-like, odorless crystals, which are stable in air. Potassium permanganate decomposes when heated at 240 Celsius with evolution of oxygen. It is not very soluble in cold water but is more soluble in warm or hot water. It reacts with alcohol and many other organic solvents, and hence, is a powerful oxidizer. Potassium permanganate reacts with hydrochloric acid liberating chlorine, and it forms explosive mixtures with all combustible materials. Solutions of potassium permanganate have an intense purple color. Handle potassium permanganate with care. Potassium permanganate is prepared from manganese ore. It is a common and commercially available chemical.

Potassium sodium tartrate



Potassium sodium tartrate forms colorless to white crystals, powder, or granules with a cooling saline taste. The compound forms a tetrahydrate, with a melting point of 90 Celsius. It becomes anhydrous when heated to 140 Celsius, and begins to decompose when heated to 220 Celsius. The crystals are soluble in water, but insoluble in alcohol and the usual organic solvents. Potassium sodium tartrate is a readily available compound. It can be made by heating potassium and sodium carbonate with tartaric acid, followed by fractional recrystallization.

Pyridine



Pyridine is colorless, flammable liquid with a characteristic disagreeable odor. It has a melting point of –41 Celsius, and a boiling point of 115 Celsius. Pyridine forms an azeotropic mixture with water boiling at 92 Celsius. It is miscible with water, alcohol, ether, oils, and many other common organic solvents. Pyridine is a weak base, but it forms salts with strong acids; it is commonly used to remove hydrogen halides from reaction mixtures. When used as a hydrogen halide scavenger, it can be recovered from reaction mixtures by filtration, and then treated with sodium carbonate to liberate the freebase pyridine, which can be collected by distillation.

Red phosphorus

$$P_4$$

Red phosphorus forms red to reddish-violet powder or granules. The crystals sublime when heated to 416 Celsius, and are insoluble in water, and most organic solvents. Red phosphorus is soluble in phosphorus tribromide. Red phosphorus is capable of converting to white phosphorus when distilled at 290 Celsius, and will ignite in air when heated to 260 Celsius. Red phosphorus can be obtained from matches, by scraping-off the match chemicals, treating these scraped-off chemicals with hot water, filtering, then treating the filtered-off solids with 99% isopropyl alcohol, allowing this alcohol mixture to soak overnight, followed by filtering-off the insoluble materials, then treating these filtered-off materials with hexane, allowing this hexane mixture to soak overnight, and then finally filtering off the red phosphorus, and then allowing it to dry. Obviously it would take a large number of matches in order to achieve a significant amount of red phosphorus, and this phosphorus may be contaminated with small amounts of glass; however, this glass does not impede in the use of the red phosphorus in anyway. Red phosphorus can be purchased from a variety of locations, so check around.

Sodium (metallic)

$$Na$$

Sodium metal forms shiny to white to lustrous granules, sticks, or cubes with a melting point of 98 Celsius. Metallic sodium readily tarnishes on exposure to air, and may react violently with moisture, so it should be stored under kerosene. Metallic sodium reacts violently and explosively with water, so avoid contact with water at all cost. Sodium can be made by electrolyzing molten sodium hydroxide using a lead anode and stainless steel cathode.

Sodium azide

$$NaN_3$$

Sodium azide forms colorless hexagonal crystals, which decompose on heating into metallic sodium and nitrogen. This decomposition often occurs violently. Sodium azide is very soluble in water, and water solutions are rapidly converted to hydrazoic acid. Sodium azide solutions should not be stored for prolonged periods of time. Sodium azide is insoluble in ether, and liquid ammonia. It is a poisonous solid, and ingestion of 600 to 800 milligrams may be fatal. Keep sodium azide in tightly sealed bottles, and store in a cool dry place away from light. It can be prepared by reacting heated nitrous oxide (60 to 90 celsius) with sodium amide heated at 60 to 100 Celsius, and then recrystallizing the sodium azide from water. Sodium azide is commercially available, but expensive.

Sodium bicarbonate

$$NaHCO_3$$

Sodium bicarbonate, also called baking soda, forms a white powder. The powder begins to lose carbon dioxide when heated to 50 Celsius, and it is converted into sodium carbonate when heated to above 100 Celsius. It is readily soluble in acid, with neutralization, but it is insoluble in alcohol and most organic solvents. A saturated sodium bicarbonate solution in water contains about 9% sodium bicarbonate by weight. The compound is obtained by bubbling carbon dioxide gas into a concentrated sodium hydroxide solution, followed by filtering-off the sodium bicarbonate. It is made on an industrial scale by bubbling carbon dioxide, or by adding dry ice to a solution of sodium chloride in ammonia.

5% Sodium bicarbonate solution: Prepare by adding and dissolving 50 grams of sodium bicarbonate into 950 milliliters of cold water.

Sodium bisulfite

$$NaHSO_3$$

or

$$Na_2S_2O_5$$

Sodium bisulfite forms a white crystalline powder with a slight odor of sulfur dioxide. The crystalline material is slowly oxidized to sodium sulfate on standing, so bottles should be kept tightly closed and stored in cool places. It is soluble in water to the extent of 1 gram in 3.5 milliliters of water, and it is only very slightly soluble in alcohol. Aqueous solutions are acidic.

Commercial sodium bisulfite contains mostly meta-sodium bisulfite (see above molecular formula; second one). Upon treating with acid, sodium bisulfite decomposes readily into sulfur dioxide gas and it is a good source of sulfur dioxide gas in the lab.

33% Sodium bisulfite solution: Prepare by adding and dissolving 100 grams of sodium bisulfite into 200 milliliters of water.

22% Sodium bisulfite solution: Prepare by dissolving 50 grams of sodium bisulfite into 170 milliliters of warm water.

20% Sodium bisulfite solution: Prepare by adding and dissolving 50 grams of sodium bisulfite into 150 milliliters of warm water.

10% Sodium bisulfite solution: Prepare by adding and dissolving 100 grams of sodium bisulfite into 900 milliliters of water.

Sodium borohydride

$$NaBH_4$$

Sodium borohydride forms hygroscopic cubic crystals, which slowly form a dihydrate when exposed to moist air. The crystals may slowly decompose when exposed to moist air, so keep bottles tightly closed and stored in a cool dry place. Sodium borohydride is soluble in alcohol (reacts slowly with alcohol at room temperature), tetrahydrofuran (reacts slowly at room temperature), and DMF. Sodium borohydride has a melting point of 37 Celsius. It can be made by reacting methyl borate with sodium hydride under inert atmosphere and moderate temperatures (80 to 120 celsius).

Sodium carbonate

$$Na_2CO_3$$

Anhydrous sodium carbonate is also called *Solway soda*, or *soda ash*. It is a odorless, hygroscopic powder with a melting point of 851 Celsius (begins to lose carbon dioxide when heated at 400 Celsius forming sodium oxide). Anhydrous sodium carbonate absorbs moisture from the air. It is soluble in glycerol and water, but insoluble in alcohol. It decomposes by acids with violent liberation of carbon dioxide. It combines with water evolving heat, and water solutions are strongly alkaline. The monohydrate forms odorless, small crystals or crystalline powder, which becomes anhydrous when heated at 100 Celsius. It is soluble in water and glycerol, but insoluble in alcohol. The decahydrate also called *Nevite*, or *washing soda*, forms transparent crystals, with a melting point of 34 Celsius. It is soluble in water and glycerol, but insoluble in alcohol. The decahydrate occurs in nature as the minerals *thermonatrite*, and *natron*. Anhydrous sodium carbonate is a widely available commercial chemical. It can be prepared by directly heating baking soda to 300 or 400 Celsius for one hour, or from scratch by bubbling carbon dioxide gas through a solution of sodium hydroxide, followed by filtration to remove the bicarbonate (baking soda), which is then roasted at 300 to 400 Celsius for one hour.

10% Sodium carbonate solution: Prepare by slowly adding and dissolving 100 grams of anhydrous sodium carbonate into 900 milliliters of hot water.

5% sodium carbonate solution: Prepare by adding and dissolving 100 grams of anhydrous sodium carbonate into 1900 milliliters of warm water.

Sodium chloride

$$NaCl$$

Sodium chloride forms white to colorless cubic crystals with a melting point of 804 Celsius. The crystals are soluble in water and glycerol, but relatively insoluble in alcohol, ether, and most common organic solvents. Sodium chloride is by far one of the most common, cheap, and readily available salts and is available in the form of rock salt or pickling salt, which is relatively pure sodium chloride.

23% Sodium chloride solution: Prepare by dissolving 100 grams of sodium chloride into 330 milliliters of warm water.

Sodium cyanide

$$NaCN$$

Sodium cyanide forms a colorless to white solid or fused granules. It is odorless, but develops a slight odor of hydrogen cyanide on standing. Sodium cyanide is a violent poison, and ingestion of as little as 50 milligrams may be fatal. Sodium cyanide has a melting point of 563 Celsius. It is freely soluble in water, but only slightly soluble in alcohol. The aqueous solution is strongly alkaline, and tends to decompose. Sodium cyanide solutions readily dissolve gold and silver forming complexes. Sodium cyanide is prepared on an industrial scale from the oxidation of methane with ammonia over a catalyst at 1000 Celsius, followed by absorption of the hydrogen cyanide into sodium hydroxide. Sodium cyanide is readily available, and can be purchased from many chemical suppliers. Avoid ingestion, and skin absorption. Sodium cyanide can be prepared by neutralizing freshly prepared liquid hydrogen cyanide (prepared from potassium or sodium ferrocyanide and dilute sulfuric acid), with sodium hydroxide or carbonate, and then recovering the desired sodium cyanide by recrystallization.

Sodium dichromate

$$Na_2Cr_2O_7$$

Sodium dichromate forms reddish to orange crystals containing 2 parts of water. The crystals become anhydrous when heated to 100 Celsius. Sodium dichromate is soluble in water, but insoluble in most organic solvents. It is a strong oxidizer, and should be kept away from combustible materials and reducing agents. Sodium dichromate is readily available from a number of sources.

Sodium hydroxide

$$NaOH$$

Sodium hydroxide forms fused solid pieces, granules, rods, or powder. It rapidly absorbs moisture and carbon dioxide from the air. Solutions of sodium hydroxide are very corrosive to animal tissue, and aluminum. It has a melting point of 318 Celsius. Sodium hydroxide is very soluble in water and alcohol. It generates large amounts of heat when dissolving in water, or when mixed with acid. Sodium hydroxide is toxic. Handle sodium hydroxide with care. Sodium hydroxide is a widely available commercial chemical, which is sold under a variety of names such as "Lye". Sodium hydroxide is prepared on an industrial scale in a procedure called the "chloro-alkali" process. In the chloro-alkali process, a sodium chloride solution is electrolysized in a special cell composed of two compartments separated by a porous membrane. Chlorine gas is produced at the positive anode, and sodium hydroxide forms at cathode.

Process for the preparation of sodium hydroxide

Summary: Sodium hydroxide can be prepared by electrolyzing a sodium chloride solution in a two-compartment cell separated by a porous membrane. Chlorine gas is liberated at the positive anode and hydrogen and sodium hydroxide are liberated at the cathode. *Use proper ventilation when running the electrolysis cell because of chlorine and hydrogen gas evolution. Run the cell in an area that is away from direct sunlight.*

Hazards: Chlorine gas is produced in this procedure; either properly vent the gas, or neutralize it by bubbling it through a sodium hydroxide or sodium carbonate solution. Carryout this procedure away from direct sun-light, and keep any source of ignition away— hydrogen gas is very flammable and explosive.

Procedure: Prepare the cell shown in the following illustration, and then add 500 grams of table salt (sodium chloride preferable sold under the name "pickling salt") to a beaker and then add 1500 milliliters of water. Then stir the mixture to dissolve the table salt. After which, pour 1000 milliliters of water into the apparatus cathode compartment. Then pour about 100 milliliters of the sodium chloride solution into the cathode compartment to bring its total volume to about 1100 milliliters. Afterwards, pour the rest of the sodium chloride solution into the apparatus anode compartment. Then put the graphite electrodes in place and electrolysis at 12-amp/12-volt until no more chlorine gas is evolved. When no more chlorine is evolved, stop the electrolysis. Then pour the cathode liquid into a beaker, and then filter to remove any insoluble materials. After filtering, pour the sodium hydroxide solution into a clean stainless steel beaker and then boil-off the water until dry sodium hydroxide solid remains. Do not use glass when boiling-off the water because the sodium hydroxide will corrode the glass and cause it to break.

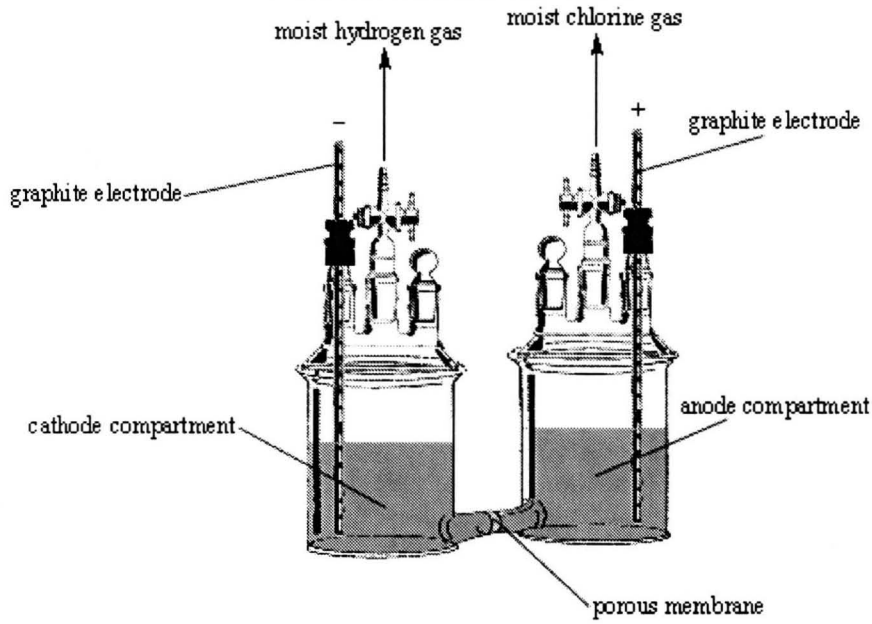


Figure 038. Apparatus for the production of sodium hydroxide. Chlorine gas is liberated at the positive anode electrode, and hydrogen gas is liberated at the negative cathode electrode. The sodium hydroxide is formed at the negative cathode electrode and remains dissolved in water.

40% Sodium hydroxide solution: Prepare by dissolving 100 grams of sodium hydroxide into 150 milliliters of cold water. Note: allow this solution to cool before using.

30% sodium hydroxide solution: Prepare by adding and dissolving 100 grams of sodium hydroxide into 230 milliliters of water. Then allow the alkaline solution to cool before using.

25% Sodium hydroxide solution: Prepare by adding and dissolving 100 grams of sodium hydroxide into 300 milliliters of cold water.

20% Sodium hydroxide solution. Prepare by adding and dissolving 100 grams of sodium hydroxide into 400 milliliters of cold water.

15% Sodium hydroxide solution: Prepare by dissolving 100 grams of sodium hydroxide into 570 milliliters of water.

10% Sodium hydroxide solution: Prepare by dissolving 100 grams of sodium hydroxide into 900 milliliters of water.

5% Sodium hydroxide solution: Prepare by adding and dissolving 50 grams of sodium hydroxide into 950 milliliters of cold water.

5% Sodium hypochlorite solution

NaOCl

5% Sodium hypochlorite, commonly called bleach, is a light yellowish liquid with a characteristic chlorine-like odor. It is a powerful oxidizing agent, and is used extensively in disinfections and decontamination procedures. It is quite stable at room temperature, but decomposes when heated forming sodium chlorate and salt. It can be easily prepared using a diaphragm cell, or by passing chlorine gas into a cold dilute sodium hydroxide solution.

Sodium iodide

NaI

Sodium iodide forms white crystalline granules or powder, with a melting point of 651 Celsius. The crystals slowly absorb moisture from the air, and slowly turn brown on exposure to light. Sodium iodide is soluble in water, alcohol, glycerol, and

slightly in acetone. The crystals should be stored in amber glass or non-clear plastic containers protected from heat and light. Sodium iodide is the chief source of iodine, and can be purchased from a number of sources—photography stores.

Sodium nitrite

$$NaNO_2$$

Sodium nitrite forms white to slightly yellow, hygroscopic granules, rods, or powder. It is very slowly oxidized to nitrate in the air. It has a melting point of 271 Celsius, and decomposes above 320 Celsius. 1 gram hexamine dissolves in 1.5 milliliters of water. It is slightly soluble in alcohol. Sodium nitrite is decomposed by acids forming brown fumes of dinitrogen trioxide. Sodium nitrite is toxic. It is a widely available commercial chemical.

Sodium sulfate

$$Na_2SO_4$$

Sodium sulfate occurs in nature as the minerals *mirabilite,* and *thenardite.* The anhydrous salt is called salt cake, and it forms a white powder or orthorhombic bipyramidal crystals. It has a melting point of 800 Celsius, and is moderately soluble in water. Sodium sulfate is insoluble in alcohol. The decahydrate is called glauber's salt, and it forms odorless, efflorescent crystals or granules, with a melting point of 32 Celsius. It becomes anhydrous when heated at 100 Celsius for 1 hour. It is soluble in water, and glycerol, but insoluble in alcohol. Sodium sulfate is a common salt of sulfuric acid, and can be made by reacting a water solution of Epsom salt with a water solution of sodium hydroxide or carbonate, and then filtering-off the precipitated by-product followed by recrystallizing the sodium sulfate from the filtered mixture. The Sodium sulfate crystals are then heated to 100 Celsius for 1 to 2 hours to form the anhydrous salt. Sodium sulfate can also be made by neutralizing a dilute sulfuric acid solution with sodium hydroxide or carbonate, and then filtering-off the precipitated by-product followed by recrystallizing the sodium sulfate from the water. The sodium sulfate crystals are then heated to 100 Celsius for 1 to 2 hours to form the anhydrous salt. Another method includes roasting anhydrous magnesium sulfate with sodium chloride, and then removing the magnesium chloride via hexane or other suitable solvent. Sodium sulfate is a widely available commercial chemical.

8% sodium sulfate solution: Prepare by adding and dissolving 100 grams of anhydrous sodium sulfate into 1150 milliliters of hot water.

Sodium thiosulfate

$$Na_2S_2O_3$$

Sodium thiosulfate forms colorless to white crystalline powder or granules. The compound readily forms a pentahydrate with a melting point of 48 Celsius. The pentahydrate looses its water of hydration when heated at 100 Celsius. Sodium thiosulfate is an effective antidote against cyanide poisoning. It can be made by mixing powdered sulfur with a 25% sodium hydroxide in 99% isopropyl alcohol solution at 0 Celsius, filtering-off the precipitated solids, and then separating the sodium thiosulfate from the sodium sulfide by fractional crystallization from an alcohol and water solution. However, sodium thiosulfate is readily available from a number of sources, and can be found at photography supply stores.

Sulfur dioxide

$$SO_2$$

Sulfur dioxide forms a colorless gas with a strong suffocating and irritating odor. The gas has a melting point of –72 Celsius, and a boiling point of –10 Celsius. The gas is easily condensed into a colorless liquid. The gas is soluble in water, alcohol, chloroform, and ether. Sulfur dioxide is available in gas cylinders, but it can be prepared in the lab by dripping 20% hydrochloric acid onto excess sodium bisulfite.

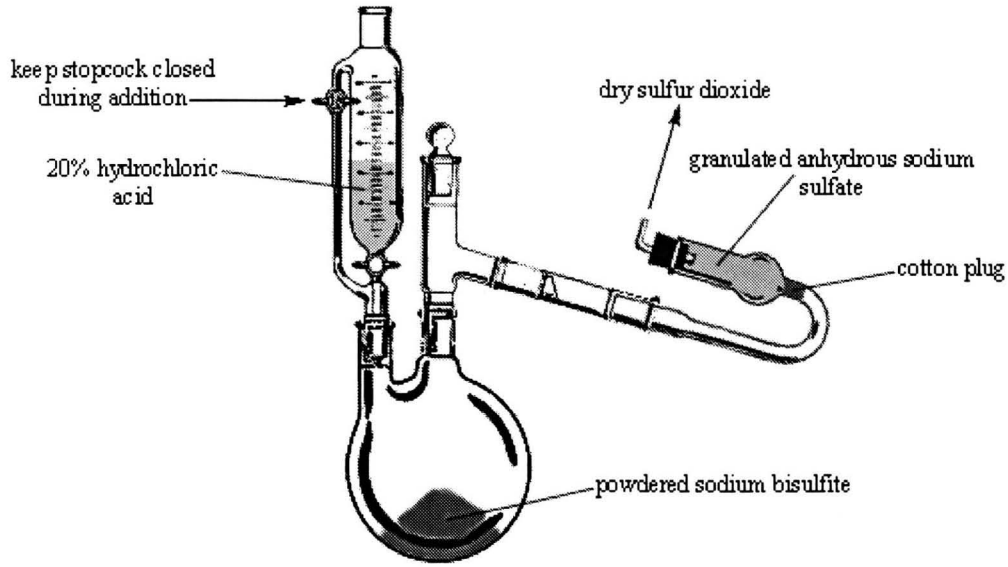
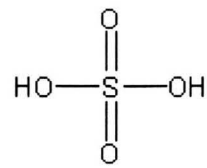


Figure 039. Set-up for the generation of sulfur dioxide gas.

98% Sulfuric acid



98% Sulfuric acid is a clear, colorless, odorless, oily liquid, which is very corrosive. 98% Sulfuric acid is referred to as concentrated sulfuric acid. Some grades of concentrated sulfuric acid may have a slight amber to brown tint due to ferrous sulfate impurity. Concentrated sulfuric acid has a great affinity for water; and hence, will char or dehydrate a great many substances. It chars wood, fabrics, resins, and also dehydrates sugar, forming carbon. Concentrated sulfuric acid has a boiling point of 290 Celsius. It decomposes when heated to 340 Celsius producing sulfur trioxide fumes and water. It has a melting point of 10 Celsius. Large amounts of heat are produced when concentrated sulfuric acid mixes with water or alcohol. When mixing with water, the acid should slowly be added. Never add the water to the concentrated acid. Concentrated sulfuric acid is a widely available commercial acid. It is the largest manufactured chemical in the world. Concentrated sulfuric acid is prepared on an industrial scale from sulfur dioxide by oxidation of sulfur or sulfides. Afterwards, the sulfur dioxide is converted into sulfur trioxide by oxidation with air over a platinum or vanadium pentoxide catalyst at 500 Celsius. The sulfur trioxide is then absorbed into 98% sulfuric acid forming 100% fuming sulfuric. This in turn is then treated with the calculated amount of water to form two parts of 98% sulfuric acid. The first part is then recycled for further absorption, and the second part is bottled and shipped. *Wear gloves when handling concentrated sulfuric acid. Concentrated sulfuric acid is a very corrosive and toxic liquid. It can cause severe skin burns and irritation. Wear proper protective clothing (certified lab coat) when handling sulfuric acid because acid spills on cloths can cause a "melting" effect of the fabric. Note: In one documented case a laboratory student working in the lab, spilled concentrated sulfuric acid onto her nylons causing the nylon fibers to "melt" to her skin. She had to be rushed to the emergency room where doctors had to surgically remove each nylon fiber.* **Note:** recovering sulfuric acid from water solutions can be accomplished by heating the sulfuric acid/water solution to 110 Celsius, and heating until no more water is evolved. This produces 93 to 98% sulfuric acid, which is perfectly suitable for use as 98% sulfuric acid (to determine when no more water can be evolved, place a piece of glass over the heated mixture. If the glass fogs-up, water is still being evolved from the sulfuric acid). **Hazards:** monitor the heating process very closely. Thick walled laboratory glass vessels should be avoided. Watch for white fumes, and be aware of potential irritating vapors; some decomposition of the acid may result.

50% Sulfuric acid solution: Prepare by dissolving 100 grams of 98% sulfuric acid into 100 grams of water. Note: allow the solution to cool before using.

25% Sulfuric acid solution: Prepare by adding and dissolving 50 grams of 98% sulfuric acid into 150 milliliters of ice cold water.

15% Sulfuric acid solution: Prepare by carefully and slowly diluting 100 grams of 98% sulfuric acid into 560 milliliters of ice water. Note: allow the solution to cool before using.

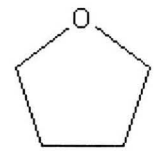
10% Sulfuric acid solution: Prepare by slowly and carefully diluting 100 grams of 98% sulfuric acid into 900 milliliters of ice water. Note: allow the solution to cool before using.

8% Sulfuric acid solution: Prepare by adding and dissolving 50 grams of 98% sulfuric acid into 570 milliliters of ice cold water.

7% Sulfuric acid solution: Prepare by diluting 50 grams of 98% sulfuric acid into 660 milliliters of cold water.

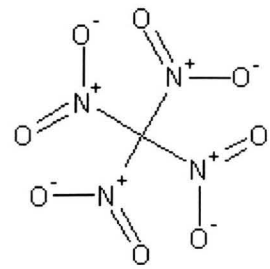
5% Sulfuric acid solution: Prepare by diluting 50 grams of 98% sulfuric acid into 950 milliliter of ice cold water.

Tetrahydrofuran. *Diethylene oxide; Tetramethylene oxide*



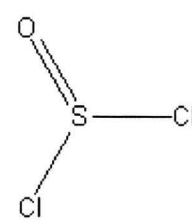
Tetrahydrofuran (THF) is a colorless liquid, with an ether-like odor. It has a melting point of –108 Celsius, and a boiling point of 66 Celsius. It is miscible with water, alcohols, acetone, ethyl acetate, ether, and hydrocarbon solvents. Tetrahydrofuran slowly forms peroxides if stored in the presence of air. For storage, fill the bottle as full as possible (leaving as little air-gap as possible). Before distilling teteahydrofuran or its mixtures, always add ferrous sulfate to check for, and destroy peroxides (a red or black color will appear). Explosions will result if peroxide contaminated tetrahydrofuran is distilled without treatment with ferrous sulfate, or other reducing agents. Tetrahydrofuran is a common solvent, and occurs in many commercial products. One source of tetrahydrofuran is PVC cement, which can be distilled to recover tetrahydrofuran. Tetrahydrofuran is a commercially available solvent.

Tetranitromethane



Tetranitromethane forms a yellowish liquid with a melting point of 14 Celsius, and a boiling point of 126 Celsius. The liquid is insoluble in water, but readily soluble in the usual organic solvents. Tetranitromethane is stable, but can form explosive mixtures when treated with bases, combustible materials, or reducing agents. The liquid attacks copper, iron, rubber, zinc, and brass, and should be stored in amber glass bottles in a cool dry place. Tetranitromethane is toxic, so avoid skin contact, inhalation and ingestion. The compound is prepared by reacting 99% nitric acid with acetic anhydride.

Thionly chloride



Thionly chloride forms a colorless to reddish fuming liquid with a melting point of –104 Celsius, and a boiling point of 76 Celsius. It decomposes rapidly with water forming hydrogen chloride, and sulfur dioxide. Thionly chloride decomposes into

chlorine, sulfur dioxide, and sulfur monochloride when heated above 140 Celsius. It is miscible with benzene, chloroform, and carbon tetrachloride. Thionly chloride is an irritating liquid, and inhalation and skin contact should be avoided. It can be made by reacting sulfur dichloride with sulfur trioxide, followed by distillation.

Tin-II-Chloride dihydrate

$$SnCl_2 \cdot 2H_2O$$

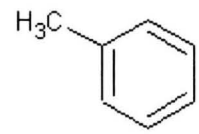
Tin-II-chloride dihydrate forms colorless to white crystals with a melting point of 38 Celsius. The crystals are slowly oxidized by air, and should be stored in airtight containers in a cool dry place. Tin-II-chloride dihydrate is soluble in water and alcohol, and very soluble in dilute hydrochloric acid. Dilute aqueous solutions of tin-II-chloride dihydrate slowly form a basic insoluble salt on prolonged standing. The compound is a powerful reducing agent, and can be made by electrolyzing a dilute solution of hydrochloric acid using a tin anode and a graphite cathode in an open cell, and then recrystallizing the dihydrate salt there from.

Tin-IV-chloride. Stannic chloride

$$SnCl_4$$

Tin-IV-chloride forms a colorless fuming liquid with a melting point of –33 Celsius, and a boiling point of 114 Celsius. The liquid is soluble in water, with formation of the pentahydrate and some decomposition. The liquid is soluble in the usual organic solvents. Tin-IV-chloride can be made by chlorinating tin at 100 to 150 Celsius, and allowing the vapors to distill over.

Toluene

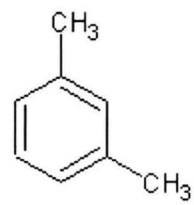


Toluene is a flammable, refractive liquid with a benzene like odor. It has a melting point of -95 Celsius, and a boiling point of 110 Celsius. Toluene is insoluble in water, but miscible with alcohol, chloroform, ether, acetone, glacial acetic acid, and carbon disulfide. Toluene is toxic. It can be obtained from tar oil, and can be found in some hardware stores.

Turpentine

Turpentine is a yellowish to brownish resinous liquid with a characteristic odor. It is insoluble in water, but readily soluble in alcohol, chloroform, ether, and glacial acetic acid. Turpentine is readily available in most hardware stores.

Xylene



Xylene is colorless liquid with a toluene like odor and a boiling point of 140 Celsius. The xylene of commerce contains a mixture of three isomers, the para, meta, and ortho isomers. Xylene is insoluble in water, but soluble in alcohol, ether, and most organic solvents. Xylene is available in most hardware stores in the solvent and paint section.

Zinc

Zn

Zinc forms bluish-white lustrous hexagonal close-packed crystals, which are stable in air. Zinc has a melting point of 419 Celsius, and a boiling point of 908 Celsius. It becomes brittle when heated to 210 Celsius, from which it can be pulverized and

ground. Finely divided zinc burns in the air forming a brilliant bluish-green flame. It is relatively stable, but reacts with acids and bases forming salts. It can be stored for years without any threat of tarnishing or discoloration, but it can be tarnished by moist air containing carbon dioxide, so it should be stored in airtight bags or containers, especially the powder or dust.

SECTION 4: AMPHETAMINES AND DERIVATIVES

Introduction

Note: the author, publisher, and any distributors, sellers, promoters, or dealers of this book take no responsibility for the actions of anyone as the result of any information in this book.

In the following section, you will learn about the preparation of a variety of amphetamines, psychedelic amphetamines, and derivatives. It should be noted that all of the compounds in this section are controlled substances with the exception of the intermediates. Be advised that any production of any of the controlled substances in this section is illegal, and subjected to federal, state, and local laws. Do not attempt to manufacture any of the following compounds unless you are qualified, trained, and are properly licensed to handle such controlled substances. Manufacture and possession of controlled substances are major offensives, and can be punishable by up to 10 years in jail—depending on the severity of the conditions of possession and/or manufacture, and the amount.

Many of the intermediates in this section are not controlled substance directly, but they fall into a special category called the "watched items" list. The watched items list is a list of chemicals, herbs, oils, and other concoctions that contain chemicals that can be used in the preparation of drugs such as for example, sassafras oil: safrole. Watched items are not directly suppressed in anyway, but are watched in the sense of orders, purchases, and the like. What this means is that safrole, for example, is a watched substance and person or persons purchasing this compound are likely to have their purchases recorded by the business from which the compound was obtained. Now purchasing safrole from an herb dealer may not be watched or recorded, but purchasing watched items from chemical suppliers definitely goes on record. These records are either instantaneously transferred to federal drug agencies (if using a credit card, or other instant payment transaction), or are transferred to federal drug agencies through the mail or by fax (if paying with checks, or cash, ect., ect.). The transfers are simple records that are filed in databases, and can be retrieved by drug agents and reviewed in the case of any suspicion or suspicious activity by the purchasers of the "watched items". Drug agents to obtain search warrants and the like can use these records, so purchasing watched items from chemical suppliers should be avoided by clandestine druggists.

For clandestine druggists, wannabe drug makers, or other "operators of illegal substances", it should be noted that your activities may be watched by law enforcement personnel, or "narcs", and manufacturing controlled substances is illegal. Before I continue, I should point out that as a chemist and writer, I cannot support, encourage or promote any illegal activities (primarily for liability sake and laws of ethics), but I will say this: if you intend to manufacture illegal drugs, please follow a few simple guidelines to keep yourself from being arrested and added to our already over populated prison system. Nevertheless, help save the taxpayers money by not manufacturing any illegal substances to begin with!

1. Never purchase chemicals in large quantities from one location. In other words, if purchasing a particular solvent (regardless whether it is readily available in hardware stores), only purchase small quantities at a time, and spread out your purchases. For example, if you need 50 gallons of a solvent, don't buy it all from one store at one time—doing so draws attention to you, and your purchase may be recorded by the store. Don't under estimate any business in this country or any other country, as they are all capable of recording and monitoring suspicious behavior. In essence, even your simple friendly local hardware store may report any suspicious purchases (even if your friends with the owners), as they are bound by law to do so. The best thing to do is purchase small amounts of your needed chemicals from several different locations.
2. Don't be stupid! Don't talk about any drugs or drug activity, don't send e-mails to friends about your activities, don't talk on cell phones and other simple to "listen in on" devices, and in short, keep your mouth closed. You would be amazed how many people have been put under from the simple fact that they opened their mouths. In these regards, silence is power, and silence can keep you out of jail. Of coarse not manufacturing controlled substance is the best method of keeping yourself out of jail.
3. If you are the druggist, and are directly responsible for the manufacture of the illegal substances, never sell the drugs directly to anyone by yourself, and always have a second person (who is not associated with you directly or the manufacture of the illegal substances in anyway). In other words, if you make the drugs, don't be the one who sells

4. them or distributes them—have someone else do the selling and distribution. Always remember, putting yourself into multiple situations increases your chances of being caught. The best thing to do is, don't be "multi-purpose", meaning stick to doing one thing, and don't conduct in more then one activity (other then your drug manufacture).
5. Use proper laboratory glassware and equipment when carrying out the preparations of the drugs. Tupperware containers, and household kitchen devices are not suitable for use in the manufacture of drugs. If you want to manufacture drugs, then it is advisable that you go out and purchase some laboratory equipment. It should be noted that laboratory equipment is not watched in anyway—this may sound surprising but it's true.
6. Clean up after yourself, and maintain a clean and proper lab. Never leave dirty glassware and other containers lying around. All laboratory equipment should be properly cleaned and then stored after each procedure. Cleaning up after yourself is only the first step, and maintaining a clean and sound working environment is the key to success. Never leave your lab intact or erected for long periods of time. In other words, don't leave things lying around, and disassemble your lab and pack it up every so often. Besides packing up your lab, it never hurts to move your lab around once in a awhile, but also remember that movement draws attention, so constantly moving your lab around should be avoided.
7. Always practice proper laboratory techniques while using proper laboratory equipment. Bad laboratory practices and poor equipment often leads to odors and other strange aromas. Many of these odors and aromas have led to the capture and arrest of clandestine druggists, and could have been avoided if these same manufactures would have practiced a few simple laboratory techniques. Odors and aromas can be avoided by using proper laboratory techniques, with proper laboratory equipment.
8. Choose your place of lab set-up properly. What this means is, don't just set-up a lab anywhere. If you live in the country, it is easier to set-up a lab and conceal it, as there are numerous places from which to establish the lab. However, even if you live in the country, suspicious activity can still draw attention to you. For example, if you set-up a lab in a shed or hole on your property somewhere, it would be very suspicious walking in an out multiple times every day or so—even if you live in the country, suspicious behavior on your own property may not go unnoticed by neighbors and the like. In other words, setting up a lab in a place that is away from the house, trailer, or place of residence should be avoided—hiding labs in make shift huts, holes, shacks, sheds, tents, ect, and hiding them behind bushes, tress or other vegetational growth is a bad idea, and leads to suspicious behavior; also, never set-up a lab in a vehicle. In essence, whether you live in the city, suburbs, or country, the best place for a lab is inside your home—perhaps in a bathroom, side room, or garage. If you have a garage that is not attached to your home, it would still be an ideal place for a lab because constantly walking in and out of this garage would not be considered suspicious as you may be working on a car or some household project. Bathrooms make excellent labs as they all come with ventilation fans, windows, and a source of water. Toilets are excellent waste receptacles, and disposal of waste chemicals can be carried out by flushing them down the toilet—of coarse the EPA would not like this. Note: carrying off waste containers to dumps, EPA waste management centers, or other waste disposal facilities is one of the worst things you could do. Never store your waste chemicals in containers, and simply dispose of any chemical waste immediately after it is generated and collected by simply flushing it down the toilet or drain—don't worry to much about environmental pollution. Most chemicals can be safely disposed of by flushing them. Avoid fires, and do not burn-off solvents and other flammable chemicals as a means of disposal. These burning chemicals produce clear smells and aromas that are not normal nor natural. Note: Even though bathrooms and other rooms may not be considered proper ventilation areas, these rooms still make excellent make shift labs, and dangerous and hazardous vapors can be dealt with by simply using proper laboratory techniques.

Notes

Laboratory glassware and equipment are readily available from many locations. You can purchase laboratory glassware from suppliers without any headaches. As previously mentioned, laboratory glassware is not controlled or watched in anyway. There are many places to find laboratory glassware and they include: on-line auction sites, hobby stores, mail order catalogs, and an arsenal of other on-line dealers. Don't just settle for what is only available in your own country, branch out and look towards other companies in other countries. In some cases, you can purchase laboratory equipment from China, for example, at rock bottom prices, and these kinds or orders are hardly ever monitored (other then any import fess or licenses). Always remember that when it comes to synthesizing drugs, nothing beats laboratory glassware and equipment—kitchen utensils and equipment are not suitable substitutes. Laboratory glassware and equipment can be expensive, but it's well worth the investment. Take care of your glassware and it will last a long time, and it will pay for itself many times over.

It should be noted that all the apparatus illustrations in this book are simply suggestive drawings, and are in no way defined as the actual needed designs. The apparatus drawings in this book are illustrated to represent a standard or suggestive design to how the apparatus should be set-up. In all consideration, apparatus design can vary, and is usually based on the availability of glassware and equipment; even though the apparatus design can vary quite significantly as can actual types of equipment used (such as plastic containers verse glass containers or glass stir rods verse stainless steel spoons, ect., ect.,), your apparatus design

should still be similar to the suggestive apparatus illustration. For example, if the suggestive apparatus illustration portrays a tube going into a reaction flask, your apparatus design should mimic this action in some form or fashion.

Many of the solvents and reagents used in the procedures in this section can be recycled. To recycle these solvents, simply place the recovered solvent layer or portion, or mixture containing the solvent, into a distillation apparatus, and collect the desired solvent by distillation. Recovered solvents should be re-distilled for quality and purity. The recovered recycled solvents should never be placed back into the storage bottles or containers that house the original fresh solvent, and the recycled solvents should be placed into separate containers. Even though this practice makes it seem like the recycled solvents may be inferior to the original fresh solvents, but this is not really the case, and storage in separate containers is a good practice to avoid any cross contamination. Recycled solvents can be used for repeating procedures, or for batch processing. Fresh solvent should be kept for separate and/or significant procedures, or for recrystallizations—note: recycled solvents used in recrystallizations can be re-used for identical future recrystallizations. It should be noted, that in any sense, most solvents can be recycled and used over and over again without compromising quality or purity of the procedures in anyway, and re-using solvents is a good practice as it saves money.

Final note: Laboratory procedures take time. In order to carryout many chemistry processes, you have to have patience and time. Most chemical procedures can take up to 24 hours or more to successfully complete, and require multiple steps. It should be noted, that chemistry is a slow subject, and in order to achieve success, you must have patience. Many of the steps involved in each procedure require multiple inner steps such as distillations, recrystallizations, and the like. Each one of these inner steps can take many hours to achieve. Distillations alone can require up to 8 hours to complete—even if it is a simple evaporation distillation. Regardless of the time needed to carryout a procedure, you need to keep in mind that this is the way things work, and there are no other means. Think of it this way, your not baking a cake—you can't simple mix a few ingredients together, bake for 2 hours at 340 degrees and wallah! Chemistry doesn't work this way. Many chemical reactions take time, and need proper conditions to work right; even with proper conditions side reactions are always a reality, and by-products always present. It's this reason that multiple steps have to be taken, and purification is usually the largest step involved in the manufacture of any given drug.

Synthetic reduction note: replacing lithium aluminum hydride

Lithium aluminum hydride is a common reducing agent used in this book to reduce nitro intermediates. However, it can be expensive, and difficult to obtain by most people. Although lithium aluminum hydride is a superior reducing agent, it can be replaced with three different techniques that work with satisfaction for reducing nitro intermediates. Lithium aluminum hydride (in this book) is primarily used for reducing nitro intermediates to the corresponding amines. These nitro intermediates are invaluable in the preparation of most amphetamines, and their reduction is crucial for the formation of amines. The reduction of these nitro intermediates can be successfully carried out using other techniques then lithium aluminum hydride without seriously compromising yields. Note: you may want to thoroughly read all the procedures in this book before reading through these reduction techniques as you may have trouble comprehending what is going on at this point in time.

A: Tin and hydrochloric acid technique

Reducing nitro intermediates with tin and hydrochloric acid is the most common method of reducing nitro compounds in the lab. The technique is generally simple, and requires little effort. The only draw back is obtaining the granulated or finely divided tin, which may be hard to acquire for some people. Nevertheless, the tin can be recycled over and over again by electrolyzing an aqueous solution of the tin chloride (recovered as a by-product of the reduction) using graphite electrodes. Tin oxide is also a by-product in the reaction, and can be converted into tin chloride by boiling with hydrochloric acid. This tin chloride can also be recycled into tin by electrolyzing an aqueous solution using graphite electrodes. It should be noted that in some cases, iron can be used, but if iron is used, only the necessary amount of concentrated hydrochloric acid should be used (preferably a 10 to 15% hydrogen chloride solution in absolute alcohol), the temperature of the reaction mixture should be kept to below 30 to 40 Celsius, and the time of reaction should be minimized to only several hours. Note: iron will not always work, and reduction can remove the amine group and replace it with a ketone group.

Into a suitable reflux apparatus (equipped with motorized stirrer or other stirring means and addition funnel), place 10 parts of your nitro intermediate, followed by 10 parts of granulated or finely divided tin, and then followed by $1/5^{th}$ part of anhydrous ferric chloride as catalyst. Thereafter, add in 50 parts of any suitable solvent selected from toluene, benzene, methylene chloride, chloroform, or hexane, and then bring the entire mixture to reflux (heat the mixture to the boiling point of the solvent, even if using a higher boiling solvent. Note: hexane is the best solvent). When the mixture begins to reflux, place 25 parts of 35 to 38% hydrochloric acid (31% muriatic acid will work) into the addition funnel, and then add in the hydrochloric acid, drop-wise over a period of about 90 minutes. During the addition, rapidly stir the reaction mixture and maintain its temperature at reflux. After adding in the hydrochloric acid, continue to reflux the entire reaction mixture for an additional 30 minutes. Thereafter, remove the heat source and allow the reaction mixture to cool to room temperature. Then drown this entire cooled

reaction mixture into 10 parts of ice water, and the stir the entire diluted reaction mixture for about 30 minutes. Thereafter, briefly extract this entire diluted reaction mixture with two 40-milliliter portions of diethyl ether, and after each extraction, discard the ether portions. Note: after each extraction, the ether will be the upper layer each time. After the extraction, place the extracted diluted reaction mixture into a beaker, and then add in a sodium hydroxide solution prepared by adding and dissolving 20 parts of sodium hydroxide into 100 parts of cold water. Note: sodium hydroxide generates excessive heat when dissolved in water, so allow the alkaline solution to cool before using. After adding in the sodium hydroxide solution, rapidly stir the entire alkaline diluted reaction mixture for about 30 minutes. Thereafter, extract this entire alkaline diluted reaction mixture with three 90-milliliter portions of diethyl ether, and after the extraction process, combine all ether portions (if not already done so), and then dry this combined ether portion, by adding to it, 15 parts of anhydrous sodium sulfate. Note: after each extraction, the ether will be the upper layer each time. After adding in the anhydrous sodium sulfate, stir the entire combined ether portion for about 10 minutes, and then filter-off the sodium sulfate. Finally, place this filtered dried ether mixture into a distillation apparatus, and distill-off the ether at 40 Celsius. When no more ether passes over or is collected, stop the distillation process, and then recover the left over remaining residue (after it has cooled). Then dissolve this left over residue into 99% isopropyl alcohol (1 part of the left over collected residue per 5 parts of 99% isopropyl alcohol), and then stir the entire alcohol mixture for about 10 minutes—thereafter, filter-off any insoluble materials (if any). Then place this filtered alcohol mixture into an ice bath, and then bubble into it, 15 parts of dry hydrogen chloride gas (excess). After the addition of the hydrogen chloride gas, add in some diethyl ether (an equal volume to the amount of isopropyl alcohol used), and then allow the entire solvent mixture to stand at 0 Celsius for about 1 hour. Thereafter, filter-off the precipitated crystals of your desired amine hydrochloride drug, and then wash these crystals with one or more small portions of diethyl ether, and then vacuum dry or air-dry the washed crystals.

B: Hydrogenation using nickel, palladium, or platinum with or without charcoal carrier

Hydrogenation is a common method of reducing nitro compounds. It is relatively simple, and relatively fast. The only draw back, although minor in most cases, is the initial investment of catalyst usually composed of palladium or platinum—both of which are quite expensive; however, all catalyst material can be recovered after the reaction and used over and over again.

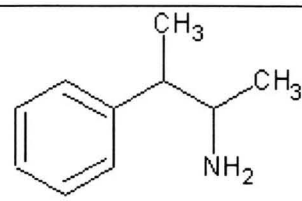
Into a suitable flask (equipped with motorized stirrer or other stirring means, and gas inlet tube), place 10 grams of your nitro intermediate, followed by 200 parts of 95% ethyl alcohol, or 99% isopropyl alcohol. Thereafter, stir the entire mixture to form a uniform mix. Then add in 10 parts of 10% hydrochloric acid in alcohol solution (prepared by bubbling 1 part of hydrogen chloride gas into 9 parts of 95% ethyl alcohol or 99% isopropyl alcohol. Note: this 10% hydrogen chloride in alcohol mixture can be obtained by extracting concentrated hydrochloric acid into alcohol, and separating the alcohol layer by adding in lots of salt—followed by lots of shaking—this process is called "Salting out". After adding in the hydrogen chloride/alcohol mixture, add in 1 part of a palladium on charcoal catalyst, or 5 parts of Raney nickel, or 1 part of a platinum gauze or sponge. Thereafter, bubble into this reaction mixture, ½ part of hydrogen gas. During the addition of the hydrogen gas, slowly stir the reaction mixture and maintain its temperature around room temperature. After the addition of the hydrogen gas, continue to stir the reaction mixture for an additional hour, and then filter-off the catalyst. This catalyst should then be briefly washed with two 25-milliter portions of 95% ethyl alcohol, and after the washings, the alcohol washing portions should be combined with the filtered reaction mixture. Then place this combined filtered reaction mixture into a distillation apparatus, and distill-off the ethyl alcohol or isopropyl alcohol (regardless of the alcohol used, heat to about 100 Celsius to drive-off the hydrogen chloride, which will carry over with the alcohol). When no more alcohol passes over or is collected, stop the distillation process, and recover the left over remaining residue (after it has cooled). Thereafter, place this recovered residue into a clean beaker, and then add in 90 parts of water, and then stir the entire mixture for about 30 minutes. After 30 minutes, add in, a sodium hydroxide solution prepared by adding and dissolving 30 parts of sodium hydroxide (excess) into 100 parts of ice-cold water. Note: sodium hydroxide generates excessive heat when dissolved in water, so allow the alkaline solution to cool before using. After adding in the sodium hydroxide, stir the entire alkaline mixture for about 1 hour at room temperature. Then, extract this entire alkaline mixture with three 90-milliliter portions of methylene chloride, and after the extraction process, combine all methylene chloride portions (if not already done so), and then dry this combined methylene chloride portion by adding to it, 10 grams of anhydrous magnesium sulfate. Note: after each extraction process, the methylene chloride will be the lower layer each time. After adding in the anhydrous magnesium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Thereafter, place this filtered dried methylene chloride portion into a distillation apparatus, and distill-off the methylene chloride at 40 Celsius. When no more methylene chloride passes over or is collected, stop the distillation process, and then collect the left over remaining residue (after it has cooled). Finally, dissolve this left over residue into diethyl ether (1 part of the left over collected residue per 5 parts of ether), and then stir the entire ether mixture for about 10 minutes—thereafter, filter-off any insoluble materials (if any). Then place this filtered ether mixture into an ice bath, and then bubble into it, 15 parts of dry hydrogen chloride gas (excess). After the addition of the hydrogen chloride gas, allow the entire ether mixture to stand at 0 Celsius for about 1 hour. Thereafter, filter-off the precipitated crystals of your desired amine hydrochloride drug, and then wash these crystals with one or more small portions of diethyl ether, and then vacuum dry or air-dry the washed crystals.

C. Reduction of the nitro intermediates with sodium borohydride

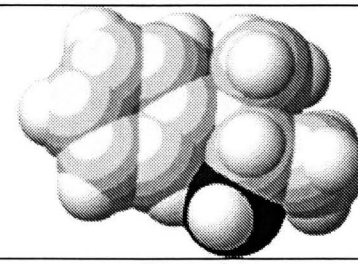
Sodium borohydride is another common reducing agent used in the reduction of nitro compounds, and yields are usually good. Sodium borohydride reduces nitro compounds in a similar manner to lithium aluminum hydride, but without the reactivity of lithium aluminum hydride.

Into a suitable reflux apparatus, equipped with motorized stirrer or other stirring means, and thermometer, place 8 parts of your nitro intermediate, followed by 120 parts of methyl alcohol. Thereafter, stir the entire mixture to form a uniform mix. Thereafter, bring this mixture to a mild reflux by heating to about 64 Celsius. When the mixture begins to gently reflux, slowly and carefully add in, in small portions (through the top of the reflux condenser) 5 parts of sodium borohydride over a period sufficient to keep the reaction mixtures temperature below 70 Celsius. During the addition of the sodium borohydride, rapidly stir the reaction mixture and maintain its temperature below 70 Celsius. After the addition of the sodium borohydride, continue to reflux and stir the reaction mixture at 64 Celsius for about 3 hours. After 3 hours, remove the heat source, and allow the reaction mixture to cool to room temperature. Thereafter, pour this entire reaction mixture into a suitable sized beaker, and then slowly and carefully add in 100 parts of ice water. Then stir the entire aqueous reaction mixture for about 1 hour. After 1 hour, add in a sodium hydroxide solution, prepared by adding and dissolving 30 parts of sodium hydroxide into 100 parts of ice water. Note: sodium hydroxide generates excessive heat when dissolved in water, so allow the alkaline mixture to cool to room temperature before using. After adding in the sodium hydroxide, stir the entire alkaline mixture for about 1 hour at room temperature. Then, extract this entire alkaline mixture with three 90-milliliter portions of methylene chloride, and after the extraction process, combine all methylene chloride portions (if not already done so), and then dry this combined methylene chloride portion by adding to it, 10 grams of anhydrous magnesium sulfate. Note: after each extraction process, the methylene chloride will be the lower layer each time. After adding in the anhydrous magnesium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Thereafter, place this filtered dried methylene chloride portion into a distillation apparatus, and distill-off the methylene chloride at 40 Celsius. When no more methylene chloride passes over or is collected, stop the distillation process, and then collect the left over remaining residue (after it has cooled). Finally, dissolve this left over residue into diethyl ether (1 part of the left over collected residue per 5 parts of ether), and then stir the entire ether mixture for about 10 minutes—thereafter, filter-off any insoluble materials (if any). Then place this filtered ether mixture into an ice bath, and then bubble into it, 15 parts of dry hydrogen chloride gas (excess). After the addition of the hydrogen chloride gas, allow the entire ether mixture to stand at 0 Celsius for about 1 hour. Thereafter, filter-off the precipitated crystals of your desired amine hydrochloride drug, and then wash these crystals with one or more small portions of diethyl ether, and then vacuum dry or air-dry the washed crystals.

0001. 2-Phenyl-3-aminobutane (freebase). *1-methyl-2-phenylpropylamine*



Structure



3D model

2-Phenly-3-aminobutane forms a colorless to slightly colored mobile liquid with an amine like odor, and acrid burning taste. It is similar to amphetamine in its toxicity, CNS stimulation, rate of effects, and detoxification. However, it is more effective then amphetamine in fighting symptoms of fatigue, and depression. It has a boiling point of 90 Celsius under a vacuum of 14 millimeters of mercury, and it is soluble in alcohol and ether with low solubility in water—the liquid is highly soluble in acids forming acid addition salts. **Note: This is a controlled substance (stimulant) as listed in the US code of Federal regulations.**

Toxicity: Low	**Rate of onset (average):** Moderate
Stimulation dosage (ingestion): 50 milligrams	**Duration of stimulation (average):** 6 to 8 hours
Stimulation dosage (inhalation): 5 to 15 milligrams	**Habit forming potential:** High
Stimulation dosage (injection): 5 milligrams +	**Estimated value U.S. (based on procedure):** $18 per gram

Procedure A: Preparation of 2-phenyl-3-aminobutane

Materials:

1. 100 grams of sodium bisulfite	9. 50 grams of anhydrous aluminum chloride

2. 72 grams of methyl ethyl ketone	10. 50 grams of diethyl ether
3. 65 grams of potassium cyanide	11. 50 milliliters of dry chloroform
4. 470 milliliters of 35 to 38% hydrochloric acid	12. 25 milliliters of 98% sulfuric acid
5. 450 milliliters of dry acetone	13. 8 grams of sodium azide
6. 113 grams of acetic anhydride	13. 200 milliliters of 10% sodium carbonate
7. 300 milliliters of 95% ethyl alcohol	14. 225 milliliters of diethyl ether
8. 160 grams of dry benzene	

Summary: 2-Phenyl-3-aminobutane can be prepared in a 3-step process starting with the formation of the cyanohydrin of methyl ethyl ketone. This cyanohydrin pre-intermediate is prepared by condensing methyl ethyl ketone with potassium cyanide in the presence of sodium bisulfite. The resulting oily liquid is removed and then converted to α,β-dimethyl hydrocinnamic acid by treating the cyanohydrin pre-intermediate with concentrated hydrochloric acid and then refluxing this existing mixture to oxidize the cyanohydrin into the methyl ethyl glycolic acid. The methyl ethyl glycolic acid is recovered by extraction with acetone, reduced to an acrylic acid derivative intermediate (not shown in the reaction equations), and then converted to α,β-dimethyl hydrocinnamic acid by treatment with dry benzene and anhydrous aluminum chloride at 45 Celsius for 3 days. Thereafter, ice and concentrated hydrochloric acid is added to the resulting mixture, and the benzene layer is then recovered, and then evaporated to yield a residue. This residue is then extracted with ether and benzene to remove the α,β-dimethyl hydrocinnamic acid from impurities, which is then recovered by evaporation of the ether/benzene. The α,β-dimethyl hydrocinnamic acid is then converted into the desired 2-phenyl-3-aminobutane by reaction with sodium azide in the presence of concentrated sulfuric acid. The resulting reaction mixture is then refluxed for several hours under mild heat, and then treated with ice and sodium carbonate. The resulting reaction mixture is then extracted with ether, and the resulting solution is then filtered, and then evaporated to remove the ether and recover the liquid product of 2-phenyl-3-aminobutane. Note: for related information, see serial number 255,882, February 11th, 1939 by Felix Haffner, and Fritz Sommer, both of Berlin Germany, to Allen property custodian.

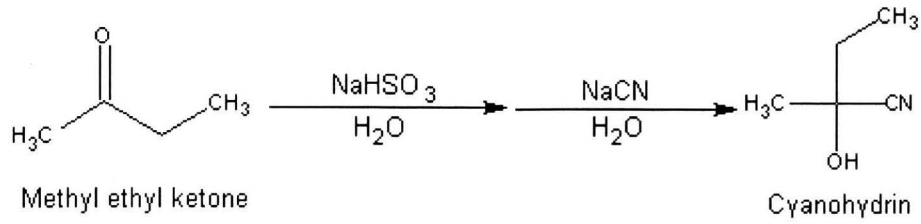
Hazards: Use caution when handling potassium cyanide, sodium azide, and benzene, all of which are toxic. Methyl ethyl ketone, acetone, and diethyl ether are highly flammable, and all form explosive mixtures with air. Perform the peroxide test before using diethyl ether that has been in storage for sometime. Use care when handling concentrated hydrochloric acid and concentrated sulfuric acid, both of which cause severe burns. Anhydrous aluminum chloride reacts violently with water, so use caution.

Procedure:

Personnel notes for procedure A: 2-phenyl-3-aminobutane

Step 1: Preparation of the cyanohydrin of methyl ethyl ketone

Prepare a solution by adding and dissolving 100 grams of sodium bisulfite into 150 milliliters of water. Note: If the sodium bisulfite fails to completely dissolve, never mind it. Thereafter, place 72 grams of methyl ethyl ketone into a suitable flask, and then place this flask into a cold water bath. Then slowly add to the methyl ethyl ketone, the sodium bisulfite solution drop wise, over a period sufficient to keep the methyl ethyl ketone below 10 Celsius. During the addition, moderately stir the methyl ethyl ketone. After the addition, stop stirring, and allow the entire mixture to stand for 10 minutes until it sets to a solid like mass. Thereafter, prepare a cyanide solution by adding and dissolving 65 grams of potassium cyanide into 100 milliliters of water, and then add this cyanide solution to the methyl ethyl ketone/sodium bisulfite mass over a period sufficient to keep the entire reaction mixture below 20 Celsius. During the addition, moderately stir the reaction mixture, and after the addition of the cyanide solution, allow the reaction mixture to stand for 10 minutes, whereby the cyanohydrin product will separate as a colorless oil. To recover this oil, place the entire reaction mixture into a seperatory funnel, and remove the lower oily cyanohydrin layer. The yield will be about 100 grams.



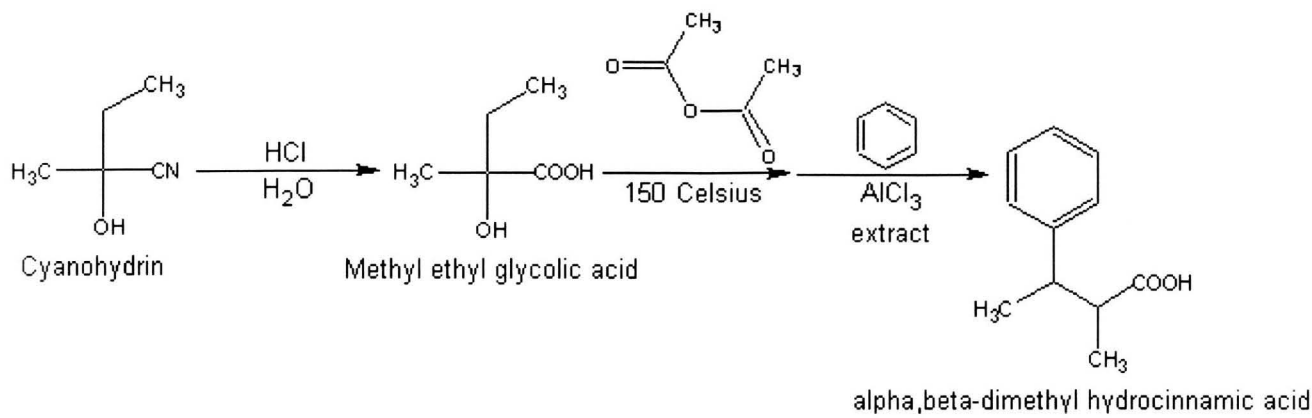
Methyl ethyl ketone — Cyanohydrin

Step 2: Conversion of the cyanohydrin into α,β-dimethyl hydrocinnamic acid

Into a suitable reflux apparatus, place all 100 grams of the cyanohydrin oily product (obtained in step 1), and then add 320 milliliters of 35 to 38% hydrochloric acid. Thereafter, reflux the entire mixture at 50 to 60 Celsius for 2 hours with constant stirring. After 2 hours, remove the heat source and allow the reaction mixture to cool to room temperature. Thereafter, extract the entire reaction mixture with three 150 milliliter portions of dry acetone, and after the extraction combine all extraction portions (if not already done so), and then place these combined acetone extracts into a distillation apparatus, or vacuum distillation apparatus and remove the acetone (preferably under vacuum). Once all the acetone has been removed, remove the dry solid product composed of methyl ethyl glycolic acid.

Next, place 90 grams of the methyl ethyl glycolic acid into a suitable flask (equipped with motorized stirrer and thermometer), and then add 113 grams of acetic anhydride. Thereafter, attach this flask to a distillation apparatus, and then heat the entire mixture for 4 hours at 150 Celsius with constant stirring. Note: During the heating process, acetic acid will be steadily evolved, and will distill over. After 4 hours, remove the heat source, and allow the reaction mixture to cool to room temperature. Thereafter, remove the remaining residue from the distillation flask, and then extract this residue with three 100-milliliter portions of 95% ethyl alcohol. After the extraction process, combine all ethyl alcohol extracts (if not already done so), and then place the combined ethyl alcohol extracts into a distillation apparatus, or vacuum distillation apparatus, and remove the ethyl alcohol (preferably under vacuum). When the alcohol has been removed, recover the acrylic acid derivative intermediate, which should have a weight of about 60 grams.

Finally, place 15 grams of the recovered acrylic acid derivative intermediate into a suitable reflux apparatus (equipped with a motorized stirrer, and thermometer) and then add 110 grams of dry benzene. Thereafter, stir the mixture to fully dissolve the acrylic acid derivative intermediate. Then add in 50 grams of finely powdered anhydrous aluminum chloride, and then reflux and stir the reaction mixture at 45 Celsius for 3 ½ days. After 3 ½ days, remove the heat source and allow the reaction mixture to cool to room temperature. Then add to the reaction mixture, 100 grams of crushed ice, followed by 150 milliliters of 35 to 38% hydrochloric acid. Then stir the entire mixture for 30 minutes at room temperature, and then place this mixture into a seperatory funnel, and remove the upper benzene layer (after removing the lower water layer first). Thereafter, place this upper benzene layer into a distillation apparatus or vacuum distillation apparatus, and remove the benzene. When no more benzene passes over, remove the heat source, and then collect the remaining residue. Then extract the desired α,β-dimethyl hydrocinnamic acid from the collected residue by adding the collected residue to a solvent mixture prepared by mixing 50 grams of diethyl ether with 50 grams of benzene. Then stir the entire mixture for 30 minutes at room temperature, and then quickly filter-off any insoluble materials. Thereafter, remove the ether and benzene using a distillation apparatus or vacuum distillation apparatus until dry solid remains. When dry solid remains, recover it from the distillation apparatus—it should weight about 15 grams.

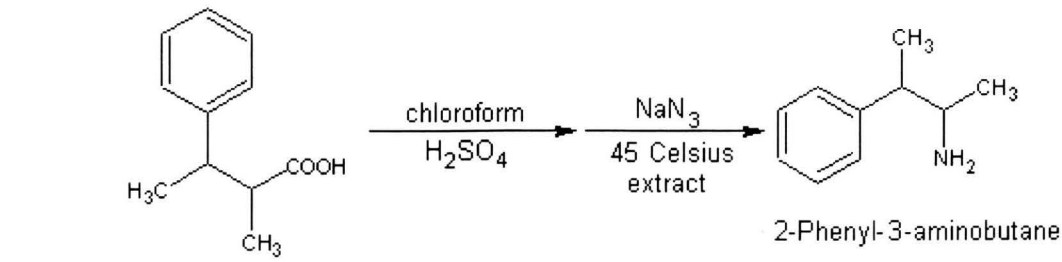


Cyanohydrin — Methyl ethyl glycolic acid — alpha,beta-dimethyl hydrocinnamic acid

Step 3: Conversion of α,β-dimethyl hydrocinnamic acid into 2-phenyl-2-aminobutane

Into a suitable reflux apparatus, place 13 grams of α,β-dimethyl hydrocinnamic acid (obtained in step 2), followed by 50 milliliters of dry chloroform. Then add in 25 milliliters of 98% sulfuric acid, and gently and carefully add in 8 grams of sodium

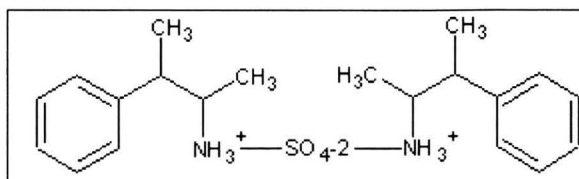
azide in small portions over a period of time as to keep the reaction mixtures temperature below 50 Celsius. Note: the addition of the sodium azide is exothermic. During the addition of the sodium azide, rapidly stir the reaction mixture. After the addition of the sodium azide, reflux the entire reaction mixture at 45 Celsius for 2 ½ hours with constant stirring. Note: During the reflux period, an evolution of gas will take place. After refluxing the reaction mixture for 2 ½ hours, remove the heat source, and allow the reaction mixture to cool to room temperature. Thereafter, pour the entire reaction mixture onto 50 grams of ice contained in a suitable beaker, and then carefully add in 200 milliliters of 10% sodium carbonate. Then stir the entire mixture until the ice melts. After the ice has melted, extract the entire mixture with three 75-milliliter portions of diethyl ether. After the extraction process, combine all ether extracts (if not already done so), and then quickly filter the combined ether extracts, and then place the filtered combined ether extracts into a distillation apparatus, or vacuum distillation apparatus, and remove the ether (preferably under vacuum). After the ether has been removed, collect the remaining liquid, which will have a boiling point of 90 Celsius under a vacuum of 14 millimeters of mercury. Note: This product can be vacuum distilled at 90 Celsius under 14 millimeters of mercury to obtain a pure product of 2-phenyl-3-aminobutane of 99%+ purity, but this is not necessary under most conditions—as this compound is generally administered as an acid addition salt.



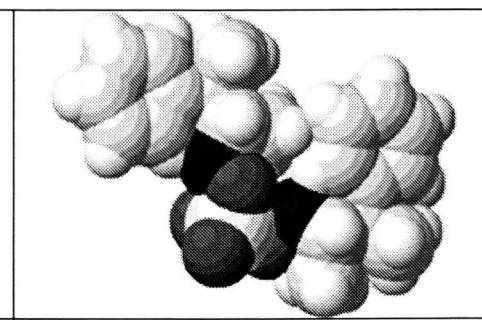
alpha,beta-dimethyl hydrocinnamic acid

2-Phenyl-3-aminobutane

0001-02. 2-Phenyl-3-aminobutane sulfate



Structure



3D model

2-Phenyl-3-aminobutane sulfate is a standard acid addition salt of the free base compound. The sulfate forms colorless to whitish crystals with a melting point of 280 Celsius. The sulfate is more soluble in water then the freebase, and can be used in making sprays, inhalers, and the like. The sulfate is more active then the freebase, and fewer quantities are needed to achieve similar effects. **Note: This is a controlled substance (stimulant) as listed in the US code of Federal regulations.**

Toxicity: Low	**Rate of onset (average):** Above moderate
Stimulation dosage (ingestion): 25 to 30 milligrams	**Duration of stimulation (average):** 6 to 10 hours
Stimulation dosage (inhalation): 5 milligrams	**Habit forming potential:** High
Stimulation dosage (injection): 5 milligrams +	**Estimated value U.S. (based on procedure):** $16 per gram

Procedure A: Preparation of 2-phenyl-3-aminobutane sulfate

Materials:

1. 15 grams of 2-phenyl-3-aminobutane	2. 5 grams of 98% sulfuric acid

Summary: The sulfate salt of 2-phenyl-3-aminobutane is readily prepared by mixing the freebase with a sulfuric acid solution. The resulting solution is then concentrated in a desiccator. Note: Similar acid addition salts can be prepared in an identical manner by substituting the sulfuric acid with 1 equal mole amount of either citric acid, d-tartaric acid, hydrochloric acid, of phosphoric acid. All acid addition salts have similar properties, toxicities, and rates of action.

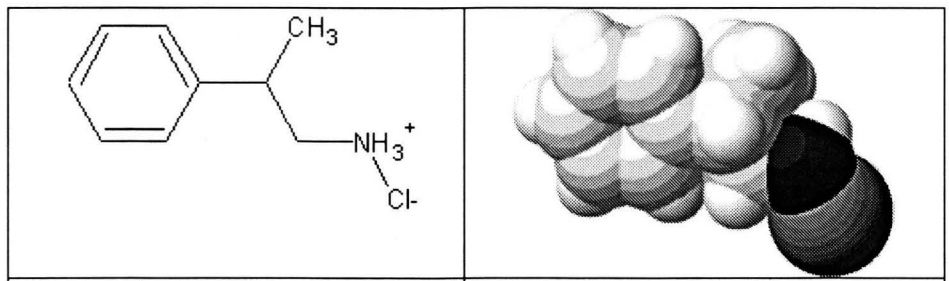
Procedure:

Personnel notes for procedure A: 2-Phenyl-3-aminobutane sulfate

Into a suitable beaker place 15 grams of 2-phenyl-3-aminobutane (prepared in 0001), and then prepare a sulfuric acid solution by adding and dissolving 5 grams of 98% sulfuric acid into 5 milliliters of water. Note: allow the sulfuric acid solution to cool to room temperature before using—as much heat is evolved upon mixing sulfuric acid with water. Thereafter, add in all at once, the sulfuric acid solution to the 2-phenly-3-aminobutane, and then stir the entire mixture for 30 minutes. Thereafter, place this mixture into a desiccator filled with anhydrous calcium chloride, and allow the mixture to stand for several days. During this standing period, the mixture will slowly become concentrated as the calcium chloride removes the water vapor. When the mixture has been concentrated, crystals of the sulfate salt should begin to form. Allow the solution to concentrate as much as possible before filtering-off the precipitated sulfate product. Once the sulfate has been collected, vacuum dry or air-dry it. Afterwards, it is ready for use, and can be molded into tablets by mixing with cornstarch, dextrose, and other food fillers, and then dead pressing 75 to 90 milligram samples into tablets.

Note: Other salts can be formed by treating 2-phenyl-3-aminobutane with the corresponding acid in an ether/ethyl alcohol solution. For example, for the hydrochloride, dissolve the 2-phenyl-3-aminobutane (1 mole freebase per 1 mole of hydrogen chloride) into a ether-ethyl alcohol solution (1 gram of freebase into 10 parts ether/5 parts 95% ethyl alcohol, and then bubble in the hydrogen chloride gas. For the citric acid, tartaric acid, or phosphoric acid salts, prepare the freebase solution in ether/ethyl alcohol as for in the hydrogen chloride example, but use 2 moles freebase per 1 mole of tartaric acid, and 3 moles of freebase per 1mole of phosphoric acid or citric acid. For each case, the ether/alcohol mixture should then be evaporated, but only evaporated to the point where 80% of the total volume is reduced. The resulting ether/alcohol concentrate can then be filtered to recover the precipitated product. All the salts are very powerful stimulants and are preferred over the freebase compound.

0002. beta-Methylphenylethylamine hydrochloride. *2-phenylpropan-1-amine hydrochloride*



Structure	3D model

beta-Methylphenylethylamine hydrochloride forms colorless to light brownish crystals with a melting point ranging from 180 to 195 Celsius (depending on purity). The crystals are moderately soluble in water, and relatively insoluble in ether. The hydrochloride salt is very similar to amphetamine hydrochloride and is an isomer to amphetamine. The stimulation effects, rate of action, and duration are almost identical to amphetamine. **Note: This is a controlled substance (stimulant) as listed in the US code of Federal regulations.**

Toxicity: Low	**Rate of onset (average):** Above moderate
Stimulation dosage (ingestion): 20 to 35 milligrams	**Duration of stimulation (average):** 6 to 8 hours
Stimulation dosage (inhalation): 6 to 8 milligrams	**Habit forming potential:** High
Stimulation dosage (injection): 5 milligrams +	**Estimated value U.S. (based on procedure):** $16 per gram

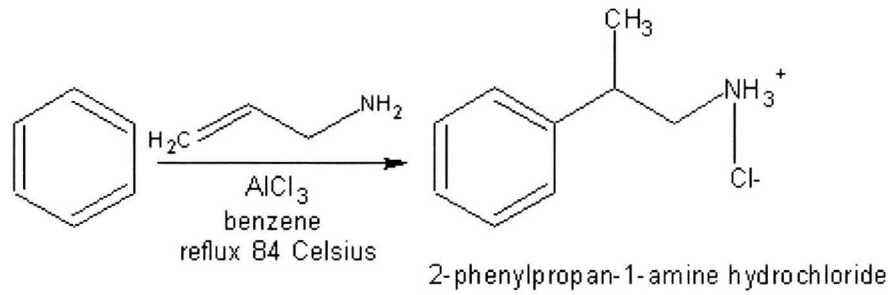
Procedure A: Preparation of beta-methylphenylethylamine hydrochloride

Materials:

1. 40 grams of anhydrous aluminum chloride	5. 305 milliliters of diethyl ether
2. 39 grams of dry benzene	6. 100 milliliters of a 30% sodium hydroxide solution

3. 5.7 grams of allylamine	7. 3.3 grams of dry hydrogen chloride gas
4. 10 milliliters of dry benzene	

Summary: beta-Methylphenylamine hydrochloride is prepared by reacting allylamine with benzene in the presence of anhydrous aluminum chloride as catalyst. The reaction is generally simple, and the product is recovered by several separations, extractions with ether, and treatment with sodium hydroxide to make the product mixture alkaline in order to remove any hydrochloride that may have formed. The product layer is then separated, and then carefully treated with dry hydrogen chloride gas to form the corresponding hydrochloride salt, which is recovered in the usual manner. Note: for related information, see serial number 609,028, August 4th, 1945 by Chester M. Suter, of Albany N.Y., and Arthur W. Weston, of Waukegan Ill., assigned by Sharp & Dohme, Inc.



2-phenylpropan-1-amine hydrochloride

Hazards: Use caution when handling diethyl ether, which is highly flammable and can form explosive mixtures with air. Use proper ventilation when handling allylamine, which is an irritant and inhalation may cause nausea, vomiting, or headache. Use proper ventilation when handling benzene, and avoid inhalation, as it is a suspected carcinogen.

Procedure:

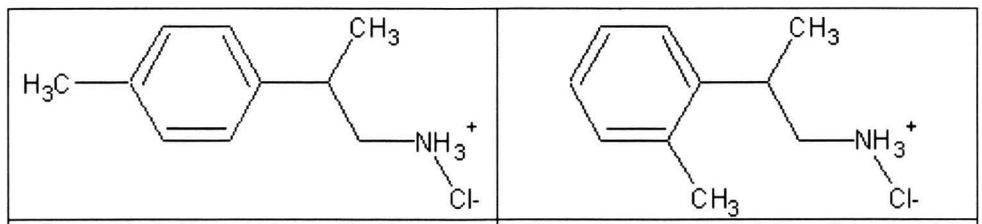
Personnel notes for procedure A: beta-methylphenylethylamine hydrochloride

Into a suitable 3-neck flask equipped with a motorized stirrer, thermometer, and addition funnel, place 40 grams of anhydrous aluminum chloride, followed by 39 grams of dry benzene. Then place this flask into an ice bath and chill to about 5 Celsius. Thereafter, prepare a solution by adding and dissolving 5.7 grams of allylamine into 10 milliliters of dry benzene, and then place this solution into the addition funnel. Then slowly add the allylamine/benzene solution to the aluminum chloride/benzene mixture over a period sufficient to keep the reaction mixture below 10 Celsius. During the addition, constantly stir the reaction mixture. After the addition of the allylamine/benzene solution, remove the ice bath, and allow the reaction mixture to warm (do not exceed 50 celsius). During this warming period, stir the reaction mixture constantly for about 30 minutes. Thereafter, replace the addition funnel with a reflux condenser, and then reflux the reaction mixture at 84 Celsius for about 3 hours. After 3 hours, remove the heat source, and allow the reaction mixture to cool to room temperature over night (whereby a brownish coloration will take place). The following day, pour the reaction mixture into a suitable sized beaker, and then add in 100 grams of crushed ice. After the ice has melted, place the entire mixture into a suitable seperatory funnel, and then remove the upper benzene layer (after removing the lower water layer first). The benzene layer can be discarded or recycled. Now, quickly mix 25 milliliters of diethyl ether with the lower water layer and swirl the total contents for about 1 minute (this is to remove the brownish color imparted to the lower water layer). Then place the total mixture into a clean seperatory funnel, and remove the upper ether layer (after removing the lower water layer first). The ether layer can be discarded or recycled. Now, place the lower water layer (which should be much clearer now), into a suitable sized beaker, and then slowly add in an excessive amount (100 milliliters of a 30% sodium hydroxide solution), to this water layer, and stir the mixture during the addition and after the addition for 10 minutes. During the addition of the sodium hydroxide solution add in small portions (no more then 100 grams) of crushed ice to the water layer to keep its temperature cool. After the addition of the sodium hydroxide solution, chill the entire water mixture (which should now be two layers) to about 10 Celsius. Note: a freezer works best for this chilling process. Afterwards, place the entire water mixture into a seperatory funnel, and then remove the upper product layer, and then place this upper product layer into a clean beaker for the meantime. Note: in some cases, the product layer will be the lower layer depending on how much sodium hydroxide you add. After removing the product layer, extract the water layer with three 50 milliliter portions of diethyl ether, and then combine all three layers afterwards (if not already done so), and then evaporate-off the ether (using a distillation apparatus or rotary evaporator) to obtain a small amount of additional product. Now add this

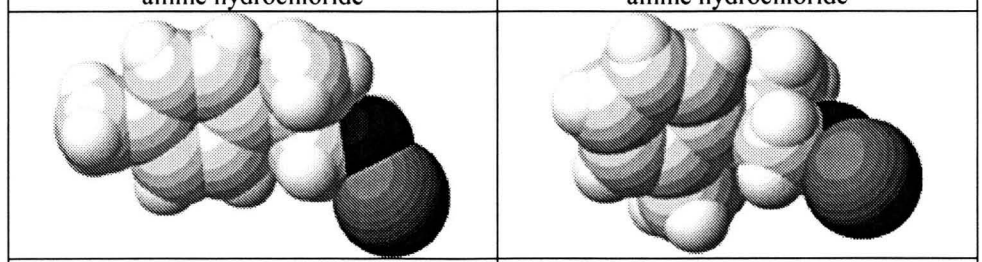
additional product to the main product layer and then dissolve the total volume into 100 milliliters of diethyl ether. Thereafter, place this ether solution of the product into an ice bath, and then slowly pass into the mixture, 3.3 grams of dry hydrogen chloride. During the hydrogen chloride addition, keep the temperature of the reaction mixture below 15 Celsius, and stir. After the addition of the hydrogen chloride, continue to stir the reaction mixture for 30 minutes at a temperature below 10 Celsius. Thereafter, filter-off any precipitated product, carefully wash this filtered-off product with 20 milliliters of diethyl ether (several times using the same 20 milliliter portion), and then vacuum dry or air dry the product. Then evaporate-off the diethyl ether from the remaining filtered mixture using a distillation apparatus or rotary evaporator to recover any dissolved product, and then wash this additional product with 10 milliliters of ether (several time using the same washing portion), and then vacuum dry or air dry the additional product.

Note: Other salts can be formed by treating beta-methylphenylethylamine freebase with the corresponding acid in an ether/ethyl alcohol solution. For example, the sulfate or tartaric acid salts can be prepared by dissolving the freebase into an ether/ethyl alcohol solution (1 gram of freebase into 10 parts ether/5 parts 95% ethyl alcohol, and then adding the desired acid (2 moles of freebase product with 1 mole sulfuric or tartaric acids). For the citric acid or phosphoric acid salts, prepare the freebase solution in ether/ethyl alcohol as previously described, but use 3 moles of freebase per 1mole of phosphoric acid or citric acid. For each case, the ether/alcohol mixture should then be evaporated, but only evaporated to the point where 80% of the total volume is reduced. The resulting ether/alcohol concentrate can then be filtered to recover the precipitated product. All the salts are very powerful stimulants and are preferred over the freebase compound.

0003. beta-Methyl-(o- and p-)methylphenylethylamine hydrochloride (mixed product)



Structure: 2-(4-methylphenyl)propan-1-amine hydrochloride	Structure: 2-(2-methylphenyl)propan-1-amine hydrochloride



3D model	3D model

beta-Methyl-(o- and p-)methylphenylethylamine hydrochloride mixture is a mixture composed of both para and ortho isomers of beta-Methyl-methylphenylethylamine hydrochloride. The substance forms whitish to off white crystals, which may have a brownish tint due to impurities, and a melting point ranging from 195 to 210 Celsius (depending on purity). The crystals are moderately soluble in water and relatively insoluble in ether. The crystals have similar properties to amphetamine in the areas of rate of action, toxicity, and duration of effects, but demonstrate fewer side effects to the more common amphetamine.
Note: This is a controlled substance (stimulant) as listed in the US code of Federal regulations.

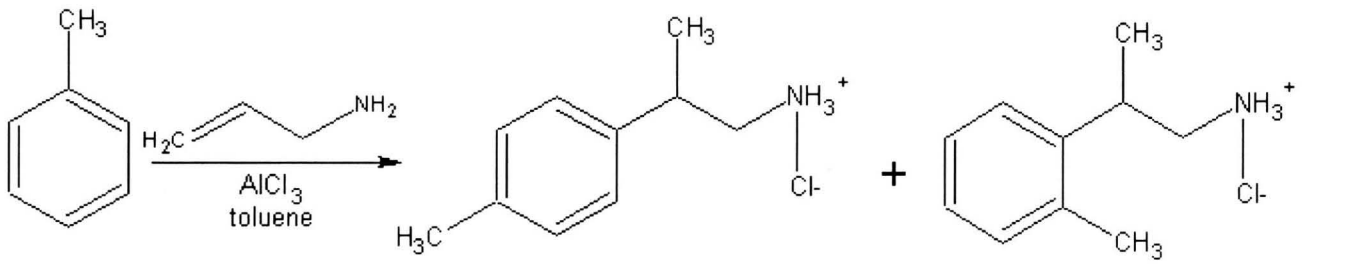
Toxicity: Low	**Rate of onset (average):** Above moderate
Stimulation dosage (ingestion): 30 to 50 milligrams	**Duration of stimulation (average):** 6 to 7 hours
Stimulation dosage (inhalation): 10 to 15 milligrams	**Habit forming potential:** Above moderate
Stimulation dosage (injection): 8 milligrams +	**Estimated value U.S. (based on procedure):** $17 per gram

Procedure A: Preparation of beta-Methyl-(o- and p-)methylphenylethylamine hydrochloride

Materials:

1. 40 grams of anhydrous aluminum chloride	5. 200 milliliters of diethyl ether
2. 46 grams of dry toluene	6. 100 milliliters of a 30% sodium hydroxide solution
3. 5.7 grams of allylamine	7. 3.2 grams of dry hydrogen chloride gas
4. 10 milliliters of dry toluene	8. 15 grams of anhydrous sodium sulfate

Summary: This mixture can be prepared in a similar fashion as beta-Methylphenylamine hydrochloride, by reacting allylamine with toluene in the presence of anhydrous aluminum chloride. The reaction is quite simple, and after the initial reaction, the reaction mixture is separated into layers (the toluene layer being discarded), and the resulting water layer of the reaction mixture is then treated with ice, then ether to remove any colored material, followed by treatment with sodium hydroxide solution. The resulting water mixture is then extracted with ether, and then treated with hydrogen chloride gas to form the hydrochloride product, which then precipitates. The filtered-off product is then washed with ether, and then dried. Note: for related information, see serial number 609,028, August 4th, 1945 by Chester M. Suter, of Albany N.Y., and Arthur W. Weston, of Waukegan Ill., assigned by Sharp & Dohme, Inc.



2-(4-methylphenyl)propan-1-amine hydrochloride

2-(2-methylphenyl)propan-1-amine hydrochloride

Hazards: Use caution when handling diethyl ether, which is highly flammable and can form explosive mixtures with air. Use proper ventilation when handling allylamine, which is an irritant and inhalation may cause nausea, vomiting, or headache. Use proper ventilation when handling toluene, and avoid inhalation.

Procedure:

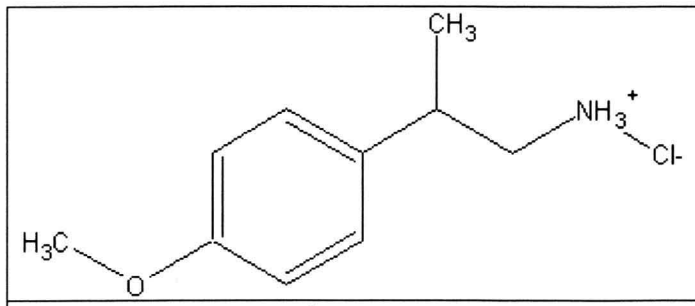
Personnel notes for procedure A: beta-Methyl-(o- and p-)methylphenylethylamine hydrochloride

Into a suitable 3-neck flask equipped with a motorized stirrer, thermometer, and addition funnel, place 40 grams of anhydrous aluminum chloride, followed by 46 grams of dry toluene. Then briefly prepare a solution by adding and dissolving 5.7 grams of allylamine into 10 milliliters of toluene, and then place this solution into the addition funnel. Thereafter, place the 3-neck flask into an ice bath, and chill the aluminum chloride/toluene mixture to about 10 Celsius. When its temperature reaches 10 Celsius, slowly add drop-wise, the allylamine/toluene solution over a period sufficient to keep the reaction mixtures temperature below 15 Celsius during the entire addition. During the addition constantly stir the reaction mixture. After the addition of the allylamine/toluene solution, remove the ice bath, and allow the reaction mixture to warm (do not exceed 50 celsius). Then allow the reaction mixture to stand overnight (do not stir). The following day, pour the entire reaction mixture into a suitable beaker, and then add in 100 grams of ice in small portions at a time. After the ice has melted, place the entire mixture into a seperatory funnel, and remove the upper toluene layer (after removing the lower water layer first). This toluene layer can be discarded or recycled. Now, quickly mix in 20 milliliters of diethyl ether to the lower water layer, and swirl the mixture for several minutes (to remove much of the dark coloring matter). Then place the entire mixture into a seperatory funnel, and remove the lower water layer. The upper ether layer can be discarded or recycled if desired. Thereafter, place the lower water layer into a clean suitable beaker, and then add in 100 milliliters of a 30% sodium hydroxide solution. Note: during the addition of the sodium hydroxide solution, slowly add in small portions, 50 grams of crushed ice to keep the mixture cool. After the addition of the sodium hydroxide solution and ice, extract the resulting water mixture with three 50-milliliter portions of diethyl ether. After the extraction process, combine all ether portions (if not already done so), and then dry the combined ether layers, by adding in 15 grams of anhydrous sodium sulfate. Then swirl the contents for several minutes (to absorb any water), and then quickly filter-off the sodium sulfate. Thereafter, place the ether mixture into a suitable beaker, and then place this suitable beaker into an ice bath, and chill to 5 Celsius. Afterwards, bubble into the ether mixture, 3.2 grams of dry hydrogen chloride gas. Note: during the addition of the hydrogen chloride, stir the reaction mixture and maintain its temperature below 10 Celsius. After the addition of the hydrogen chloride gas, stir the mixture at 5 Celsius for 1 hour, and then filter-off any precipitated product. Then wash this filtered-off product with 20 milliliters of diethyl ether (several times using the same washing portion), and then vacuum dry or air-dry the product. The remaining filtered ether mixture can be evaporated

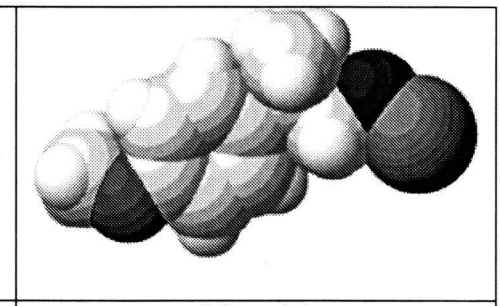
using a distillation apparatus or rotary evaporator, to recover a little more remaining product, which can then be washed with 10 milliliters of diethyl ether (several times using the same washing portion), and then vacuum dried or air dried.

Note: Other salts can be formed by treating the mixed freebase product (collected by evaporation of the ether mixture instead of treatment with dry hydrogen chloride gas) with the corresponding acid in an ether/ethyl alcohol solution. For example, the sulfate or tartaric acid salts can be prepared by dissolving the freebase into an ether/ethyl alcohol solution (1 gram of freebase into 10 parts ether/5 parts 95% ethyl alcohol, and then adding the desired acid (2 moles of freebase product with 1 mole sulfuric or tartaric acids). For the citric acid or phosphoric acid salts, prepare the freebase solution in ether/ethyl alcohol as previously described, but use 3 moles of freebase per 1mole of phosphoric acid or citric acid. For each case, the ether/alcohol mixture should then be evaporated, but only evaporated to the point where 80% of the total volume is reduced. The resulting ether/alcohol concentrate can then be filtered to recover the precipitated product. All the salts are very powerful stimulants and are preferred over the freebase compound.

0004. beta-Methyl-p-methoxy-phenethylamine hydrochloride



Structure


3D model

beta-Methyl-p-methoxy-phenethylamine hydrochloride, also called 2-(4-methoxyphenyl)propan-1-amine hydrochloride forms white to light darkened crystals with a melting point ranging from 230 to 250 celsius (depending on purity). The crystals are somewhat soluble in water, and relatively insoluble in ether. The compound can be used as an effective substitute for amphetamine, or other similar drugs. beta-Methyl-p-methoxy-phenethylamine hydrochloride is slightly more toxic then its predecessors, but it demonstrates excellent adrenalinic effects, and has a slighter faster rate of onset.
Note: This is a controlled substance (stimulant) as listed in the US code of Federal regulations.

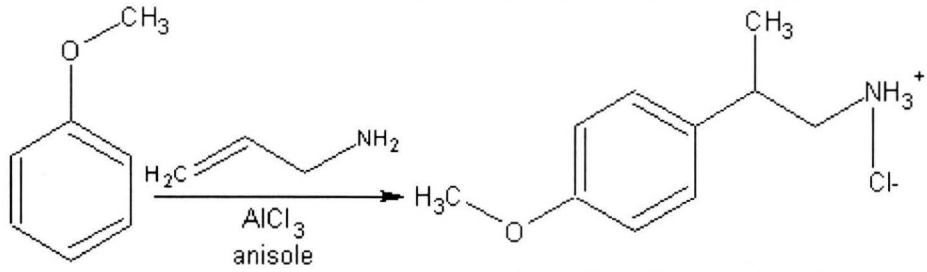
Toxicity: Moderate	**Rate of onset (average):** Above moderate
Stimulation dosage (ingestion): 50 to 60 milligrams	**Duration of stimulation (average):** 6 to 8 hours
Stimulation dosage (inhalation): 15 to 20 milligrams	**Habit forming potential:** Moderate
Stimulation dosage (injection): 10 milligrams +	**Estimated value U.S. (based on procedure):** $14 per gram

Procedure A: Preparation of beta-Methyl-p-methoxy-phenethylamine hydrochloride

Materials:

1. 40 grams of anhydrous aluminum chloride	5. 100 milliliters of a 30% sodium hydroxide solution
2. 42 grams of anisole	6. 3.5 grams of dry hydrogen chloride gas
3. 5.7 grams of allylamine	7. 15 grams of anhydrous sodium sulfate
4. 220 milliliters of diethyl ether	

Summary: beta-Methyl-p-methoxy-phenethylamine hydrochloride is readily prepared using a familiar process where in allylamine and anhydrous aluminum chloride are reacted with a phenyl compound, in this case the simple compound anisole. In essence, the anisole is reacted with allylamine in the presence of anhydrous aluminum chloride, which acts as the catalyst. The reaction is generally simple, and afterwards, the reaction mixture is treated with ice (to hydrolyze the aluminum chloride), and the resulting mixture is then briefly extracted with ether to remove the unreacted anisole solvent. The extracted reaction mixture is then treated (titrated) with sodium hydroxide solution to dissolve the insoluble aluminum salts, and the resulting reaction mixture is then extracted with ether. The ether extracts are then treated with dry hydrogen chloride gas to form the desired hydrochloride product. The product is then simply recovered by filtration, and the product is then washed, and then dried. Note: for related information, see serial number 609,028, August 4th, 1945 by Chester M. Suter, of Albany N.Y., and Arthur W. Weston, of Waukegan Ill., assigned by Sharp & Dohme, Inc.



2-(4-methoxyphenyl)propan-1-amine hydrochloride

Hazards: Use caution when handling diethyl ether, which is highly flammable and can form explosive mixtures with air. Use proper ventilation when handling allylamine, which is an irritant and inhalation may cause nausea, vomiting, or headache.

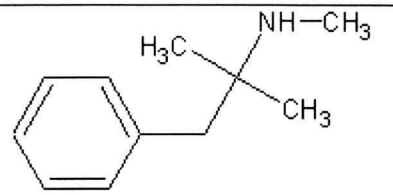
Procedure:

Personnel notes for procedure A: beta-Methyl-p-methoxy-phenethylamine hydrochloride

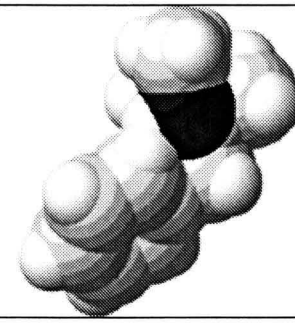
Into a suitable 3-neck flask, equipped with a motorized stirrer, thermometer, and addition funnel, place 32 grams of anisole, followed by slowly adding 40 grams of anhydrous aluminum chloride. Thereafter, place the 3-neck flask into an ice bath, and chill the anisole mixture to 5 Celsius. When its temperature reaches 5 Celsius, prepare a solution by adding and dissolving 5.7 grams of allylamine into 10 grams of anisole, and then place this solution into the addition funnel. Thereafter, slow add this allylamine/anisole solution to the anisole/aluminum chloride mixture over a period sufficient to keep the reaction mixture below 10 Celsius. During the addition, constantly stir the reaction mixture. After the addition of the allylamine/anisole solution, continue to stir the reaction at 10 Celsius for 10 hours. After 10 hours, remove the ice bath, and then pour the entire reaction mixture into a suitable sized beaker, and then add in 100 grams of crushed ice. Then allow the entire mixture to stand at room temperature overnight. The following day, briefly extract the entire mixture with two 20-milliliter portions of diethyl ether (to remove the left over anisole), and the combined ether extracts can be discarded or recycled if desired. Now, to the left over mixture after extraction, add in 100 milliliters of a 30% sodium hydroxide solution, and add in small portions of ice (50 grams) during the addition to keep the mixture cool. During the addition of the sodium hydroxide solution, stir the mixture. Note: a magnetic stirrer can be used for stirring at this point. After the addition of the sodium hydroxide solution and ice, extract the entire mixture with three 50-milliliter portions of diethyl ether. After the extraction, combine all ether portions (if not already done so), and then to these combined ether extracts, add in 15 grams of anhydrous sodium sulfate (to absorb water). Afterwards, filter-off the sodium sulfate after stirring the total mixture for several minutes to ensure proper water absorption. Thereafter, place the filtered ether mixture into a suitable beaker, and then place this beaker into an ice bath, and chill the contents of the beaker to 5 Celsius. Then bubble into the ether mixture 3.5 grams of dry hydrogen chloride gas. During the addition of the hydrogen chloride gas, constantly stir the reaction mixture. After the addition of the hydrogen chloride gas, filter-off any precipitated product, and then wash this product with 20 milliliters of fresh diethyl ether (several times using the same washing portion), and then vacuum dry or air-dry the washed product. A small amount of additional product can be obtained by evaporating-off the ether from the filtered mixture using a distillation apparatus or rotary evaporator. This additional product should then be washed with 10 milliliters of fresh diethyl ether, followed by vacuum drying or air-drying.

Note: Other salts can be formed by treating the beta-methyl-p-methoxy-phenethylamine freebase product (collected by evaporation of the ether mixture instead of treatment with dry hydrogen chloride gas) with the corresponding acid in an ether/ethyl alcohol solution. For example, the sulfate or tartaric acid salts can be prepared by dissolving the freebase into an ether/ethyl alcohol solution (1 gram of freebase into 10 parts ether/5 parts 95% ethyl alcohol, and then adding the desired acid (2 moles of freebase product with 1 mole sulfuric or tartaric acids). For the citric acid or phosphoric acid salts, prepare the freebase solution in ether/ethyl alcohol as previously described, but use 3 moles of freebase per 1mole of phosphoric acid or citric acid. For each case, the ether/alcohol mixture should then be evaporated, but only evaporated to the point where 80% of the total volume is reduced. The resulting ether/alcohol concentrate can then be filtered to recover the precipitated product. All the salts are very powerful stimulants and are preferred over the freebase compound.

0005. N-methyl-omega-phenyl-tert-butylamine. *N,2-dimethyl-1-phenylpropan-2-amine; New Ice; Extravagance.*



Structure



3D model

N-methyl-omega-phenyl-tert-butyamine forms a colorless to dark oily liquid with a sharp biting taste—all the salts form white to off white crystals (some of which may be discolored due to impurities). The freebase is a powerful stimulant, and the salts thereof are even more powerful stimulants, all of which have excellent properties. The freebase and salts thereof are more effective then amphetamine or methamphetamine, and preferred over the aforementioned for CNS stimulation. N-methyl-omega-phenyl-tert-butylamine and its salts demonstrate fewer side effects then amphetamine or methamphetamine, but have similar toxicities. Note: N-methly-omega-phenyl-tert-butyamine and its salts are highly addictive, and demonstrate analgesic activity (pain killing activity) along with its CNS stimulations. Note: alcoholic beverages may increase the effects.

Note: This is a controlled substance (stimulant) as listed in the US code of Federal regulations.

Toxicity: Low	**Rate of onset (average):** Very good
Stimulation dosage (ingestion): 35 to 50 milligrams	**Duration of stimulation (average):** 6 to 8 hours (depending on the person)
Stimulation dosage (inhalation): 10 to 20 milligrams	**Habit forming potential:** High
Stimulation dosage (injection): 10 milligrams +	**Estimated value U.S. (based on procedure):** $16 per gram

Procedure A: Preparation of N-methyl-omega-phenyl-tert-butylamine

Materials:

1. 128 grams of anhydrous aluminum chloride	13. 175 grams of potassium hydroxide
2. 850 milliliters of pure benzene	14. 70 grams of bromine
3. 92 milliliters of isobutryl chloride	15. 150 milliliters of 95% ethyl alcohol
4. 100 milliliters of 35 to 38% hydrochloric acid	16. 24 grams of finely divided calcium hydroxide
5. 300 milliliters of 5% sodium carbonate solution	17. 200 milliliters of diethyl ether
6. 150 grams of anhydrous sodium sulfate	18. 6 grams of benzaldehyde
7. 950 milligrams of anhydrous ferric chloride	19. 35 milliliters of 95% ethyl alcohol
8. 475 milliliters of anhydrous liquid ammonia	20. 5 grams of methyl iodide
9. 10.05 grams of metallic sodium	21. 30 milliliters of methanol
10. 350 milliliters of dry toluene	22. 33 milliliters of a 15% acetic acid solution
11. 38 grams of benzyl bromide	23. 100 milliliters of a 30% sodium hydroxide solution
12. 125 milliliters of petroleum ether	

Summary: N-methyl-omega-phenyl-tert-butylamine is prepared in a multiple step process starting with the formation of isobutyrophenone. This isobutyrophenone is prepared by reacting isobutryl chloride with benzene in the presence of anhydrous aluminum chloride as catalyst. After the reaction, the reaction mixture is treated with concentrated hydrochloric acid, and the benzene layer is then recovered. The remaining reaction mixture is then extracted with benzene, and all benzene portions are then combined, and then evaporated to recover the residual product. This residual product, is then converted into 1,3-diphenyl-2,2-dimethyl-propanone-1 by reaction with sodamide (prepared by the interaction of metallic sodium with liquid ammonia), followed by treatment with benzyl bromide in the presence of benzene. Thereafter, the reaction mixture is drowned into cold water, and the solvent layer then recovered. The residual product of 1,3-diphenyl-2,2-dimethyl-propanone-1 is then recovered by evaporation of the reaction mixture. The 1,3-diphenyl-2,2-dimethyl-propanone-1 is then converted into alpha-alpha-dimethyl-beta-phenylpropionamide by reaction with sodamide in toluene at 60 Celsius. The resulting reaction mixture is then cooled, drowned into water, and the organic layer then removed. This organic layer is then concentrated by distillation or by evaporation, and the resulting concentrated organic solution is then treated with petroleum ether to induce crystallization of the desired alpha-alpha-dimethyl-beta-phenylpropionamide. This product is then converted into di-(beta-phenyl-alpha,alpha-di-methylethyl) urea by treating the alpha-alpha-dimethyl-beta-phenylpropionamide product with potassium hypobromite in the presence of excess water. The resulting reaction mixture is then heated for several hours at 60 Celsius, and then the resulting mixture is boiled to drive-off the excessive amount of water. When dry solid remains, the dry solid mix is then extracted with

95% ethyl alcohol to dissolve the desired product. The desired product of di-(beta-phenyl-alpha,alpha-di-methylethyl) urea is then recovered from the ethyl alcohol mixture by recrystallization. The desired product of di-(beta-phenyl-alpha,alpha-di-methylethyl) urea is then converted into omega-phenyl-tert-butylamine by reaction with finely divided calcium hydroxide in the presence of a small amount of water. During the reaction, the desired product is simultaneously distilled over. After the reaction period, the distilled product is collected, and then extracted with ether to dissolve the desired product. The desired product is then collected by evaporation of the ether. The desired product of omega-phenyl-tert-butylamine is then converted into N-methyl-omega-phenyl-tert-butylamine by condensation with benzaldehyde followed by treatment with methyl iodide under heat and pressure. The resulting mixture is then refluxed with methanol, and then titrated with acetic acid followed by hydrolysis with a sodium hydroxide solution. The resulting mixture is then extracted with ether, and the resulting ether mixture is then evaporated to recover the desired product as a colorless to dark colored oily liquid. Purification under high vacuum results in the pure desired product of N-methyl-omega-phenyl-tert-butylamine as a colorless oil. For similar information, see serial number 775,754 September 23rd, 1947 by Liese L. Abell, of New york City, William F. Bruce, of Havertwon, Pa, and Joseph Seifter, of Willow Grove, Pa., to Wyeth Incorporated.

Hazards: Extinguish all flames before using benzene, toluene, ether, ethyl alcohol, methanol, and petroleum ether. Ether can form explosive mixtures with air. Use great care when handling metallic sodium, and avoid skin contact, and contact with most chemicals. Use care when handling concentrated hydrochloric acid, which evolves toxic and corrosive fumes. Where gloves when handling benzyl bromide, which is an irritant, and handle sodium hydroxide, anhydrous aluminum chloride, potassium hydroxide, and isobutryl chloride with care—all of which can cause skin irritation. Use care when handling liquid ammonia, which is corrosive and very irritating to the eyes, nose, and throat.

Procedure:

Personnel notes for procedure A: N-methyl-omega-phenyl-tert-butylamine

Step 1: Preparation of isobutyrophenone

Into a suitable 1000-milliliter 3-neck flask equipped with motorized stirrer, thermometer, addition funnel, and reflux condenser, place 128 grams of anhydrous aluminum chloride. Thereafter, quickly add in 200 milliliters of benzene (the benzene should be as pure as possible), and thereafter prepare a solution by adding and dissolving 92 milliliters of isobutryl chloride into 100 milliliters of pure benzene, and then place this solution into the addition funnel. Then slowly add drop wise the isobutryl chloride/benzene solution to the contents of the 3-neck flask. During the addition, the contents in the 3-neck flask (the reaction mixture) should be constantly stirred during the entire addition. Note: during the addition, the temperature of the reaction mixture will rise—never mind this. After the addition of the isobutryl chloride solution, reflux the entire reaction mixture at 84 Celsius for 90 minutes with moderate stirring. After refluxing for 90 minutes, remove the heat source, and allow the reaction mixture to cool to room temperature. Thereafter, pour the entire reaction mixture into a suitable sized beaker, and then rapidly add in 100 milliliters of 35 to 38% hydrochloric acid, and immediately thereafter, quickly add in 500 grams of crushed ice. Then stir the entire mixture for 30 minutes, and then decant (pour off) the upper benzene layer, or use a seperatory funnel to remove the upper benzene layer (after removing the lower water layer first). Note: keep this upper benzene layer. Once the upper benzene layer has been removed, extract the lower water layer with three 150-milliliter portions of benzene. After the extraction process, combine all benzene extracts (if not already done so), and then add this combined benzene extract with the previous upper benzene layer. Thereafter, wash the total volume of benzene with three 100-milliliter portions of 5% sodium carbonate solution, followed by three 150-milliter portions of water. Note: After each washing portion, the benzene will be the upper layer each time, and can be recovered by simple decantation, or using a seperatory funnel in the usual manner. After the washing process, add in to the total volume of benzene, 50 grams of anhydrous sodium sulfate (to absorb water), and then stir the entire mixture for 10 minutes. Thereafter, filter-off the sodium sulfate, and then place the mixture into a distillation apparatus, or rotary evaporator, and remove all the benzene. When no more benzene passes over or is collected, stop the distillation or evaporation process, and remove the remaining residue that is left behind, after it has cooled. This residue will contain predominantly the desired product of isobutyrophenone, and can be purified by high vacuum distillation at 81 Celsius under a vacuum of 1 millimeter of mercury. However, this high vacuum purification process is not always necessary.

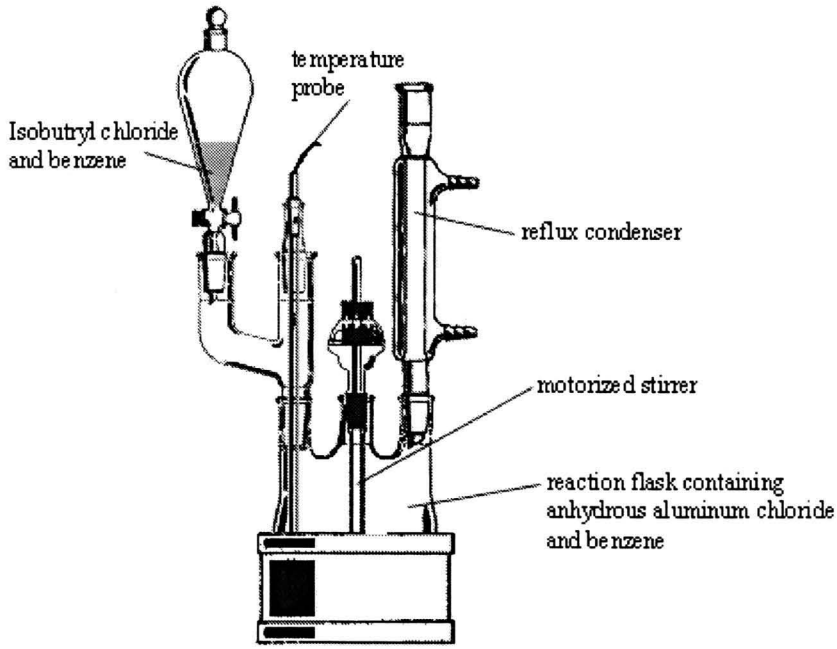


Figure 040. Reflux apparatus used in step 1.

Step 2: Preparation of 1,3,-diphenyl-2,2-dimethyl-propanone-1

Into a suitable 3-neck flask equipped with distillation adapter, a thermometer, motorized stirrer, and addition funnel, place 500 milligrams of anhydrous ferric chloride, followed by 300 milliliters of anhydrous liquid ammonia, and then quickly add in 6.25 grams of metallic sodium. Then stir the entire mixture for about 10 minutes. Thereafter, evaporate-off the liquid ammonia using an appropriate distillation apparatus equipped with a dry ice condenser so the ammonia vapor can be condensed back into a liquid and recycled for further crops. Once the liquid ammonia has been removed, add to the previous 3-neck flask (containing the left over residue after removal of the ammonia) 100 milliliters of dry toluene, and then immediately thereafter, slowly add in drop wise (over a period of about 60 minutes), a solution prepared by adding and dissolving 37 grams of the product obtained in step 1, and 38 grams of benzyl bromide into 100 milliliters of dry benzene. Note: this solution should be prepared before adding the metallic sodium to the liquid ammonia. During the addition of the solution, stir the reaction mixture constantly. After the addition of the solution, reflux the entire reaction mixture at 84 Celsius for 24 hours. After refluxing the reaction mixture for 24 hours, remove the heat source, and allow the reaction mixture to cool to room temperature. Then pour the entire reaction mixture into a suitable sized beaker, and then add in 350 milliliters of cold water, and then stir the entire mixture for 30 minutes. Thereafter, remove the upper organic liquid layer (either by decantation or by a seperatory funnel), and then to this upper organic layer, add in 50 grams of anhydrous sodium sulfate (to absorb water), and then stir the entire mixture for 10 minutes. Then filter-off the sodium sulfate, and then place the filtered organic liquid into a distillation apparatus, or rotary evaporator, and remove the organic solvents (mostly toluene, and benzene). When all the solvents have been removed, recover the remaining residue left behind, and then place it aside for step 3. Note: this left over residue can be purified by high vacuum distillation at 142 Celsius under a vacuum of 3 millimeters of mercury. However, this purification process is not always necessary.

Step 3: Preparation of alpa-alpha-dimethyl-beta-phenylpropionamide

Into a 3-neck flask, equipped with a distillation apparatus fitted with a dry ice condenser, thermometer, motorized stirrer, and addition funnel, place 175 milliliters of anhydrous liquid ammonia, followed by 450 milligrams of anhydrous ferric chloride, followed by 3.8 grams of metallic sodium. Thereafter, stir the entire mixture for about 30 minutes, and then evaporate-off the excess liquid ammonia (the dry ice condenser is used to recover the ammonia in liquid form so it can be recycled). After removal of the liquid ammonia, add to the remaining left over residue, 125 milliliters of dry toluene, and then reflux this mixture at 60 Celsius. Then prepare a solution by adding and dissolving 35.7 grams of the product obtained in step 2 into 125 milliliters of dry toluene. Note: this solution should be prepared before adding the metallic sodium to the liquid ammonia. When the reaction mixture reaches a temperature of 60 Celsius, slowly add in drop wise, the previously prepared solution (the product obtained in step 2 dissolved in toluene), over a period of about 60 minutes while rapidly stirring the reaction mixture. After the addition of the said solution, continue to heat and stir the entire reaction mixture at 60 Celsius for 3 hours. After 3 hours, remove the heat source, and then allow the reaction mixture to cool to room temperature. Then pour the entire reaction mixture into a suitable sized beaker, and then add in 250 milliliters of cold water, and stir the entire mixture for 30 minutes.

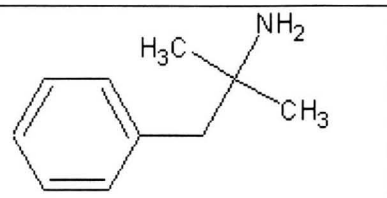
Thereafter, remove the upper organic layer (either by decantation or by using a seperatory funnel), and then to this upper organic layer, add in 50 grams of anhydrous sodium sulfate and then stir the entire mixture for 10 minutes. Afterwards, filter-off the sodium sulfate, and then place the entire filtered organic layer into a distillation apparatus or rotary evaporator, and evaporate-off the solvents until the total volume of the mixture is cut in half (meaning concentrate the organic layer by evaporation). When the total volume of the organic layer has been concentrated to half its original volume, stop the distillation and/or evaporation process, and then pour the remaining contents into a beaker (allow it to cool before removing from the apparatus). Then place this beaker into an ice bath, and then to the concentrated organic layer in the beaker, add in 125 milliliters of petroleum ether, whereby the desired product will slowly crystallize out of solution. Note: during the previous concentrating process, some of the desired product may have already crystallized out. If this is the case, never mind it. After the addition of the petroleum ether, allow the entire mixture to stand in the ice bath for several hours. Thereafter, filter-off the precipitated product (crystallized product), and then vacuum dry or air this participated product. Note: this desired product can be purified by recrystallization from a solvent mixture prepared by mixing 150 milliliters of dry benzene to 175 milliliters of petroleum ether, but this is not necessarily needed.

Step 4: Preparation of di-(beta-phenyl-alpha,alpha-di-methylethyl) urea

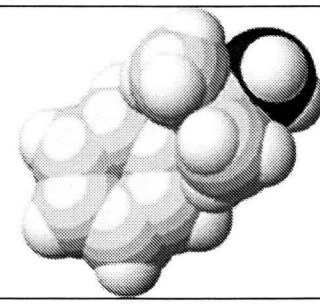
Prepare a solution (solution A) by adding and dissolving 7 grams of the product obtained in step 3 into 840 milliliters of water. Then prepare a second solution (solution B), by adding 175 grams of potassium hydroxide into 700 milliliters of water (note: potassium hydroxide generates much heat when dissolved in water, so allow the solution to cool before proceeding any further). Once the potassium hydroxide solution has cooled, add in 70 grams of bromine, and stir the mixture during the addition (potassium hypobromite will form). Thereafter, add solution A to solution B over a period of about 30 minutes while stirring solution B. After the addition of solution A to solution B, heat the entire reaction mixture at 60 Celsius, for about 4 hours. Note: a reflux apparatus is not needed and the reaction mixture can be heated at 60 Celsius in a beaker. Thereafter, boil the entire reaction mixture at 100 Celsius while stirring to drive-off the water. Note: a magnetic stirrer/hot plate can be used to stir the reaction mixture. When solid products begin to form (after much of the water has been driven-off, reduce the heat to about 95 to 98 Celsius, and carefully drive-off the remaining water until nothing but dry solids remain. When dry solids remains, thoroughly mix the entire dry solids with 150 milliliters of 95% ethyl alcohol (after the dry solids have cooled to room temperature). Thereafter, recover the ethyl alcohol mixture by filtering-off any undissolved solids, and then recrystallize the desired product form this ethyl alcohol mixture (simply evaporate-off or distil-off the majority of the ethyl alcohol, or simply boil-off all the ethyl alcohol until dry solid remains. Afterwards, recover the product, and vacuum dry or air-dry it if necessary, and then set it aside for step 5.

Step 5: Preparation of **omega-phenyl-tert-butylamine**

Into a suitable sized distillation apparatus, equipped with a thermometer, place 6 grams of the product obtained in step 4, and then add in 30 milliliters of water, followed by 24 grams of finely divided calcium hydroxide. Thereafter, heat the entire mixture to 240 Celsius for 90 minutes. During the 90 minute heating process, the desired product, as well as the water will distill over. After 90 minutes, remove the heat source, and then recover the distilled components contained in the receiver flask of the distillation apparatus. Then place the recovered components into a beaker, and then mix well with 50 milliliters of diethyl ether to dissolve the desired product). Thereafter, place this ether mixture into a distillation apparatus or rotary evaporator, and evaporate-off the ether to recover the desired product. After the ether has been removed, recover the remaining residue, which should be a semi-solid to liquid mass, and place aside for step 6. Note: this desired product can be purified by distillation under vacuum at 84 Celsius under a vacuum of 9 millimeters of mercury, but this is not necessarily needed.



Structure



3D model

Note: this product can be used as a stimulant, and is similar to the main product discussed in this procedure. Note: This is a controlled substance (stimulant) as listed in the US code of Federal regulations.

Toxicity: Low	**Rate of onset (average):** Very good
Stimulation dosage (ingestion): 45 to 50 milligrams	**Duration of stimulation (average):** 6 to 8 hours (depending

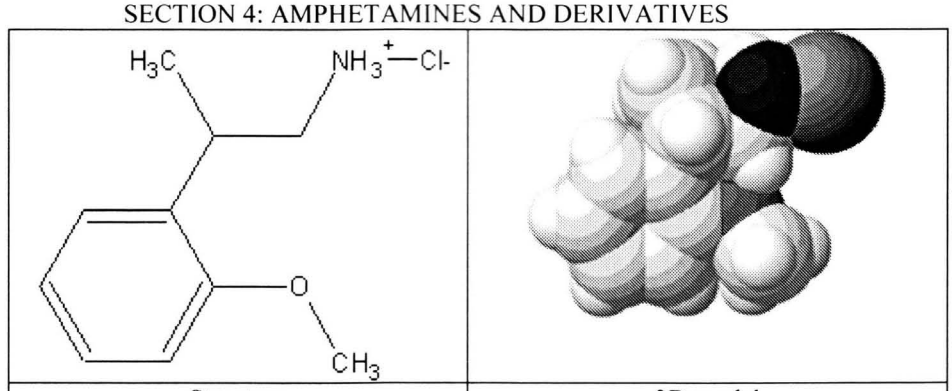
	on person)
Stimulation dosage (inhalation): 10 to 20 milligrams	**Habit forming potential:** High
Stimulation dosage (injection): 10 milligrams +	**Estimated value U.S. (based on procedure):** $12 per gram

Step 6: Preparation N-methyl-omega-phenyl-tert-butylamine

Prepare a solution into a reflux apparatus by adding 8 grams of the product obtained in step 5, followed by 6 grams of benzaldehyde, followed by 35 milliliters of 95% ethyl alcohol. Thereafter, reflux this entire mixture for 15 minutes at 78 Celsius. After the reflux period, replace the reflux condenser, with a conventional condenser, fitted to a receiver flask, and then conventionally distill the reaction mixture at 80 Celsius to drive-off the ethyl alcohol. Once the ethyl alcohol has been distilled-off, recover the left over residue remaining (after it has cooled to room temperature), and then place this left over residue aside for just a moment. Then to this left over residue, weigh out 8 grams of it, and then place 8 grams of this residue into a suitable sized flask, and then add in 5 grams of methyl iodide. Now, place a large, tightly fitting balloon over the flask, and then heat the contents in this flask to about 90 Celsius for 6 hours. Note: The balloon is placed over the flask not only to create an airtight seal, but to create pressure as the contents are heated. When the contents in the flask are heated, the pressure will cause the balloon to inflate. In most cases, a metal circular screw clamp should be used to securely fasten the balloon to the flask. During the heating process, the balloon will inflate and expand, but should not blow. This inflation and expansion will produce enough pressure inside the flask to properly carryout the reaction of the methyl iodide with the residue. If the balloon does happen to blow or break, quickly replace it with another one, and continue the heating process for the remaining amount of time. After heating the residue with the methyl iodide for 6 hours at 90 Celsius. Remove the heat source and allow the contents in the flask to cool to room temperature. Note: during this cool down, watch the balloon so it does not get sucked into the flask due to backpressure. When the contents have cooled to room temperature, remove the balloon, and then place the entire contents of the flask into a beaker, and then add in 30 milliliters of methanol, followed by 3 milliliters of water. Then place this mixture into a reflux apparatus, and reflux at 68 Celsius for 15 minutes. Afterwards, remove the heat source, and allow the mixture to cool to room temperature. Then pour this mixture into a beaker, and then quickly add in 33 milliliters of a 15% acetic acid solution. Immediately thereafter, boil this solution at 100 Celsius for 50 minutes. After 50 minutes, remove the heat source, and allow the mixture to cool to room temperature. Then add in 100-milliliters of a 30% sodium hydroxide solution, and then stir the entire mixture for 1 hour. Finally, extract this alkaline mixture with three 50-milliliter portions of diethyl ether, and then combine all portions of the ether once finished. Then place the combined ether extracts into a distillation apparatus, or rotary evaporator, and remove the ether. When no more ether passes over, stop the evaporation process, and allow the left over contents to cool to room temperature, before removing said contents. The resulting contents will be the desired product of N-methyl-omega-phenyl-tert-butylamine (freebase). If desired, this product can be purified by high vacuum distillation at 75 Celsius under a vacuum of 1 millimeter of mercury to obtain a colorless oil. **Note:** the pressure process discussed above, whereby a balloon is placed over a flask, can be substituted by using a conventional steel pipe with threads at both ends. To carryout the steel pipe technique, pour all necessary materials (as describe for the balloon technique), into a thick walled stainless steel pipe, and then seal both ends with the corresponding steel caps. The threads at each end should be wrapped with Teflon tape prior to screwing in the end caps. Then place the entire pipe, and submerge it into a water bath and heat at the desired temperature for the desired time. Note: this process can be dangerous and can lead to pressure explosions. Carryout the process in an area that can contain any such explosion, and maintain a safe distance away during the operation—just to be on the safe side.

Note: Salts of the omega-phenyl-tert-butylamine and N-methyl-omega-phenyl-tert-butylamine, such as the hydrochloride, sulfate, tartrate, citrate, and phosphate can be prepared by dissolving the desired product into ether (1 gram product per 15 milliliters of ether), and then adding the corresponding acid (for the hydrochloride, hydrogen chloride gas should be bubbled into the ether mixture rather then by adding hydrochloric acid. For hydrochloric acid, 1 part of the acid gas per 1 part of the freebase product dissolved in the ether should be bubbled into the ether mixture followed by evaporation of the ether to recover the entire hydrochloride product. For sulfuric acid or tartaric acid, 1 part of the acid should be added per 2 parts of the freebase product dissolved in the ether; and for citric acid or phosphoric acid, 1 part of the acid should be added per 3 parts of the freebase product dissolved in the ether. For each case, the ether should only be evaporated to the point where only 80% of the total volume is reduced. The resulting ether concentrate can then be filtered to recover the precipitated product. All the salts are very powerful stimulants and are preferred over the freebase compounds.

0006. β-o-Methoxyphenyl-n-propylamine hydrochloride. *2-(2-methoxyphenyl)propan-1-amine hydrochloride*



Structure	3D model

2-(2-methoxyphenyl)propan-1-amine hydrochloride forms white to off-white crystals with a melting point of 134 Celsius. The crystals are moderately soluble in water, and relatively insoluble in ether. The crystals can be used as a delayed action stimulant with stimulation effects similar to, but less severe then amphetamine or methamphetamine. 2-(2-methoxyphenyl)propan-1-amine hydrochloride has fewer side effects then amphetamine or methamphetamine, and does not interfere or cause any lapse in rest or sleep. It can be used in combination with alcoholic beverage to provide increased stimulation and feelings of well being.

Note: This substance is a controlled substance (stimulant) as listed in the US code of Federal regulations.

Toxicity: Moderate	**Rate of onset (average):** Low
Stimulation dosage (ingestion): 40 to 50 milligrams	**Duration of stimulation (average):** 4 to 6 hours (depending on the person)
Stimulation dosage (inhalation): 20 to 30 milligrams	**Habit forming potential:** Moderate
Stimulation dosage (injection): 10 milligrams +	**Estimated value U.S. (based on procedure):** $21 per gram

Procedure A: Preparation of β-o-Methoxyphenyl-n-propylamine hydrochloride

Materials:

1. 37 grams of 2-methoxy acetophenone	11. 90 milliliters of 35 to 38% hydrochloric acid
2. 50 grams of ethyl bromoacetate	12. 150 milliliters of petroleum ether
3. 25 grams of a copper-zinc alloy (containing 8% copper by weight)	13. 333 milliliters of a 8% sodium sulfate solution
4. 425 milliliters of dry benzene	14. 26.9 grams of sodium hydroxide
5. 39 grams of 98% sulfuric acid	15. 5 milliliters of 98% sulfuric acid
6. 80 grams of anhydrous magnesium sulfate	16. 75 milliliters of a 50% sulfuric acid solution
7. 15 milliliters of phosphorus oxychloride	17. 18 grams of thionly chloride
8. 18.6 grams of potassium hydroxide	18. 125 milliliters of a 28 to 30% ammonia solution
9. 200 milliliters of 95% ethyl alcohol	19. 12.5 grams of liquid bromine
10. 850 milliliters of diethyl ether	20. 2 grams of dry hydrogen chloride gas

Summary: β-o-Methoxyphenyl-n-propylamine hydrochloride is prepared in a six step process beginning with the formation of Ethyl-(β-methyl)-o-methoxy cinnamate. This product is prepared by reacting 2-methoxy acetophenone with ethyl bromoacetate in the presence of a zinc-copper alloy. The reaction is carefully regulated, and afterwards, the product containing layer, is removed, treated with sulfuric acid to remove any zinc and copper, and the resulting mixture is then separated, treated with phosphorus oxychloride, and then distilled to remove the benzene and recover the desired residue. The residue, which contains predominantly the desired Ethyl-(β-methyl)-o-methoxy cinnamate is then converted into ortho-methoxy-β-methyl cinnamic acid by reaction with potassium hydroxide in ethyl alcohol. The reaction is rather general, and afterwards, the reaction mixture is refluxed, diluted with cold water, and then acidified with concentrated hydrochloric acid. This resulting acidified solution is then extracted with ether to recover the desired product, which is recovered by removal of the ether. The resulting residue containing predominantly the ortho-methoxy-β-methyl cinnamic acid, is then converted into β-(ortho-methoxy phenyl) butyric acid by electrolysis using a special electro chemical cell. This electrochemical cell consists of two separate compartments, both of which are separated by a porous membrane. The reaction primarily takes place in the cathode compartment, whereby the desired product is formed. After the electrolysis procedure, the contents of the cathode compartment are recovered, and then acidified using sulfuric acid. The resulting acidified mixture is then extracted with ether, and the desired β-(ortho-methoxy phenyl) butyric acid then recovered by evaporation of the ether. The resulting residue is then recrystallized from a solvent mixture to obtain a refined product of β-(ortho-methoxy phenyl) butyric acid. This refined compound, is then converted into β-(ortho-methoxy phenyl) butryl chloride by treatment with thionly chloride under mild heat, the resulting reaction mixture is

then stripped of excess chloride, and the remaining residue is then converted into the amide by treatment with concentrated ammonia solution to form the desired β-(ortho-methoxy phenyl) butyramide, which is then collected by filtration, and the resulting collected solid product is then recrystallized from benzene to obtain a purified product. The resulting purified β-(ortho-methoxy phenyl) butyramide is then finally converted into the master product of β-ortho-methoxy phenyl-n-propylamine hydrochloride by reaction with a hypobromite solution prepared on site by the interaction between sodium hydroxide and liquid bromine. The following reaction mixture is then heated for a short period, and then extracted with ether. The ether extract is then treated with dry hydrogen chloride gas, and the resulting product then collected by filtration. For related information, see serial number: 275,638, May 25th, 1939 by Eugene H. Woodruff, from Kalamazo, Mich., to The UpJohn Company.

Hazards: Extinguish all flames before using benzene, ether, ethyl alcohol, and petroleum ether. Ether can form explosive mixtures with air. Use great care when handling phosphorus oxychloride, and thionly chloride, both of which can cause skin burns, and react violently with water evolving corrosive fumes. Use great care when handling liquid bromine, which is very irritating to the eyes, nose, and throat, and can severe skin irritation. Use care when handling concentrated hydrochloric acid, hydrogen chloride gas, and sulfuric acid, the prior evolves toxic and corrosive fumes. Where gloves when handling ethyl bromoacetate, which is an irritant, and causes irritation to the skin and respiratory tract. Handle sodium hydroxide, and potassium hydroxide with care—both of which can cause skin irritation. Use care when handling concentrated ammonia solutions, which are corrosive and very irritating to the eyes, nose, and throat.

Procedure:

Personnel notes for procedure A: β-o-Methoxyphenyl-n-propylamine hydrochloride

Step 1: Preparation of Ethyl-(β-methyl)-o-methoxy cinnamate

Into a suitable reflux apparatus (equipped with thermometer and motorized stirrer), place 37 grams of 2-methoxy acetophenone (obtained from the Sigma-Aldrich chemical company, page 1177 of the 2003-2004 Aldrich catalog), followed by 50 grams of ethyl bromoacetate (obtainable from the Sigma-Aldrich chemical company, page 841 of the 2003-2004 Aldrich catalog), followed by 25 grams of a zinc-copper alloy (containing 8% copper by weight), and finally 125 milliliters of dry benzene. Thereafter, heat the entire reaction mixture to 100 Celsius until a positive reaction begins. Note: this positive reaction will be easily observed as the reaction becomes quite violent in nature. However, before the reaction becomes violent (the positive reaction), remove the heat source and allow the reaction mixture to cool to about 50 Celsius. In other words, heat the reaction mixture to 100 Celsius for an initial period of about 10 to 15 minutes. Note: during all this, moderately stir the reaction mixture constantly. After the reaction begins, and becomes violent, and thereafter, the heat source being removed and the reaction mixture thereby cooled to 50 Celsius, reflux the entire reaction mixture at 84 Celsius for 30 minutes. After refluxing for 30 minutes, remove the heat source, and allow the reaction mixture to cool to room temperature. Thereafter, prepare a solution by adding 39 grams of 98% sulfuric acid into 150 milliliters of ice water. Note: the addition of sulfuric acid to water generates much heat. Allow the resulting sulfuric acid solution to cool to room temperature before using. Then add to the reaction mixture, this dilute sulfuric acid solution over a period of about 5 to 10 minutes while constantly stirring the reaction mixture. After the addition of the sulfuric acid, continue to stir the reaction mixture for about 30 minutes, and then filter-off any insoluble materials. Then, place the filtered reaction mixture into a seperatory funnel, and remove the upper benzene layer (which will be colored orange), after removing the lower acidic water layer first. Thereafter, add to the removed benzene layer, 25 grams of anhydrous magnesium sulfate (to absorb water), and then filter-off this magnesium sulfate only after stiring the mixture for 10 minutes. Now, into a clean reflux apparatus, place this dried benzene layer, and then gently add in 15 milliliters of phosphorus oxychloride. Then reflux the entire mixture for 30 minutes at about 84 Celsius. After refluxing for 30 minutes, remove the heat source, and allow the reaction mixture to cool to room temperature. Thereafter, pour the entire reaction mixture into a clean beaker, and then add in two 75-milliliter portions of ice-cold water, and then stir the whole mixture for 10 minutes. Then, simply remove the upper benzene layer (by decantation or by using a seperatory funnel—after removing the lower water layer first), and then add to the removed benzene layer, 25 grams of anhydrous magnesium sulfate (to absorb water), and then filter-off this magnesium sulfate after stirring the entire mixture for 10 minutes. Finally, place the filtered benzene layer into a distillation apparatus, or rotary evaporator, and remove the benzene until no more benzene passes over. When no more benzene passes over, remove the left over residue (after allowing it to cool to room temperature), and then place this residue aside for step 2. Note: This residue can be distilled at 160 Celsius under a vacuum of 13 millimeters of mercury to obtain a purified product, but this is not generally needed for step 2. The result will be about 38 grams of the desired product.

Step 2: Preparation of ortho-methoxy-β-methyl cinnamic acid

Into a suitable reflux apparatus equipped with a thermometer, place 36 grams of the product obtained in step 1, followed by a potassium hydroxide solution prepared by adding and dissolving 18.6 grams of potassium hydroxide into 19 milliliters of cold water. Note: potassium hydroxide generates much heat when dissolved in water, so allow the solution to cool before using. Next, add into the same reflux apparatus, 200 milliliters of 95% ethyl alcohol, Thereafter, reflux the entire reaction mixture to 78 Celsius for 1 hour. After refluxing for 1 hour, remove the heat source, and allow the reaction mixture to cool to room temperature. Afterwards, drown the entire reaction mixture into 750 milliliters of cold water. Then, place this entire mixture into a distillation apparatus, and remove all the ethyl alcohol. When most of the ethyl alcohol has been distilled-off, remove the heat source, and allow the remaining aqueous solution to cool to room temperature. Now, extract this remaining aqueous solution with two 50-milliter portions of diethyl ether (to remove unreacted impurities), and then after the extraction process, combine both ether layer (if not already done so), and then discard or recycle the ether extracts. Note: during each extraction portion, the ether will be the upper layer. Once the aqueous solution has been extracted with ether to remove unreacted impurities, add to the aqueous solution, 90 milliliters of 35 to 38% hydrochloric acid, and thereafter, extract this acidified aqueous solution with three 100-milliliter portions of diethyl ether. Note: after each extraction, the ether will be the upper layer. After the extraction process, combine all ether portions (if not already done so), and then place the combined ether portions into a distillation apparatus, or rotary evaporator, and remove the ether. When no more ether can be removed, collect the left over residue remaining (after it has cooled to room temperature), and then recrystallize the desired product (contained in the residue) from a solvent mixture prepared by adding and dissolving 75 milliliters of benzene into 75 milliliters of petroleum ether. After the Recrystallization process, vacuum dry or air-dry the product. The result will be about 33 grams of the desired product as crystals with a melting point of about 76 Celsius.

Step 3: Preparation of β-(ortho-methoxy phenyl) butyric acid (electrolysis process)

Into the cathode compartment of a suitable electrochemical cell (see the following figure—with two compartments divided by a porous membrane), place 32 grams of the product obtained in step 2, followed by 333 milliliters of a 8% sodium sulfate solution, and then add in 3 grams of sodium hydroxide. Then manually stir the entire mixture until all solids dissolve. Thereafter, place 350 milliliters of cold water into the anode compartment, followed by 5 milliliters of 98% sulfuric acid. Then place a mercury electrode (negative pole), into the cathode compartment, and then place a small sheet of lead (as the anode electrode) in the anode compartment. Note: however, a mercury cathode is preferred, it can be replaced by a graphite, aluminum, or zinc electrode—all of which can be in the form of a rod, usually $1/4^{th}$ inch in diameter by 6 inches long. Once the cell has been assembled, pass 6 amps worth of current (preferably 6 amps at 6 volt) through the cell for about 4 hours. Note: more then 4 hours may or may not be needed depending on the exact cathode used, and size. Note: power supplies are available through a number of places, and can be purchased as "battery chargers" from local stores. Remember that the black clamp is the negative (-), and the red clamp is the positive (+).

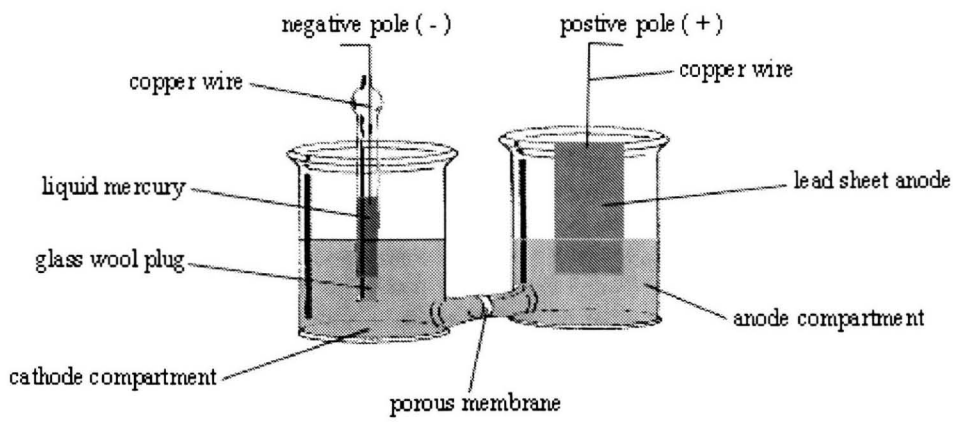


Figure 041. Electrochemical cell for step 3.

After the electrolyses process, unplug the electrical source, and then recover the contents in the cathode compartment, and then filter these contents to remove any insoluble precipitates. Thereafter, add to the filtered contents, 75 milliliters of a 50% sulfuric acid solution to precipitate the product. Note: more 50% sulfuric acid may or may not be needed to precipitate any additional product. Afterwards, instead of filtering-off any precipitated product, extract the entire acidic mixture with three 75-milliliter portions of diethyl ether (make sure that any precipitated solids are thereby dissolved into the ether). After the extraction process, combine all ether extracts (if not already done so), and then add to the combined ether portions, 15 grams of

anhydrous magnesium sulfate (to absorb water). Then stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Now, place this filtered ether mixture into a distillation apparatus, or rotary evaporator, and remove the ether. When no more ether can be removed, collect the left over residue remaining (after it has cooled to room temperature), and then recrystallize the desired product (contained in this residue) from a solvent mixture prepared by adding and dissolving 75 milliliters of benzene into 75 milliliters of petroleum ether. After the Recrystallization process, vacuum dry of air-dry the product. The result will be about 28 grams of the desired product as crystals with a melting point of about 47 Celsius.

Step 4: Preparation of β-(ortho-methoxy phenyl) butryl chloride

Into a 3-neck flask (equipped with a thermometer, addition funnel and reflux condenser) place 19.4 grams of the product obtained in step 3, and then place 18 grams of thionly chloride into the addition funnel. Then gently heat the 3-neck flask to about 40 Celsius, and when the temperature of the 3-neck flask reaches about 40 Celsius, slowly add the thionly chloride drop wise, over a period of about 15 minutes. After the addition, continue to heat the reaction mixture for about 30 additional minutes. Afterwards, replace the reflux condenser with a standard cold-water condenser fitted with a receiver flask, and then distill-off any unreacted thionly chloride at a temperature of 76 Celsius until no more liquid passes over. When no more liquid passes over or is collected, remove the heat source, and allow the remaining contents left to cool to room temperature before collecting. Once collected, set aside for step 5. Note: if desired, the collected product can be purified by vacuum distillation at 140 Celsius under a vacuum of 12 millimeters of mercury. However, this process is not really needed for step 5. The result will be 18 grams of the desired product.

Step 5: Preparation of β-(ortho-methoxy phenyl) butyramide

Into a suitable 3-neck flask equipped with thermometer, motorized stirrer, inlet tube, and a standard funnel, place 125 milliliters of a 28 to 30% aqueous ammonia solution. Then place this 3-neck flask into an ice bath, and chill to 5 celsius. When the temperature of the aqueous ammonia reaches about 5 Celsius, slowly add in small portions (via the standard funnel), 17.6 grams of the product obtained in step 4. Note: during the addition of the 17.6 grams of product obtained in step 4, moderately stir the reaction mixture and maintain its temperature below 10 Celsius at all times. Second note: also during the addition of the 17.6 grams of product obtained in step 4, pass dry ammonia gas into the reaction mixture during the entire addition in order to keep the reaction mixture saturated with ammonia. After the addition of the 17.6 grams of product obtained in step 4, stop bubbling the dry ammonia gas into the reaction mixture, and then continue to stir the reaction mixture for 15 additional minutes while keeping the reaction mixtures temperature below 10 Celsius at all times. Thereafter, filter-off the precipitated product, wash this filtered-off product with several small portions of cold water, and then vacuum dry or air-dry the filtered-off product. Then recrystallize this dried product from 150 milliliters of benzene, and after the recrystallization process, simply vacuum dry or air-dry the product. The result will be 14 grams of the desired product with a melting point of 125 Celsius.

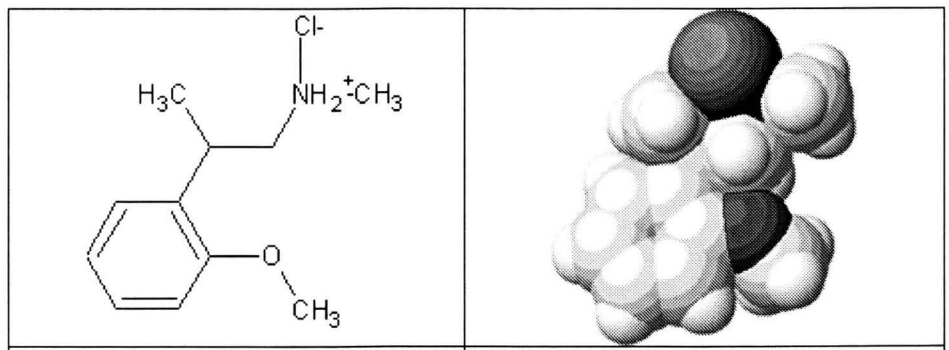
Step 6: Preparation of β-ortho-methoxy phenyl-n-propylamine hydrochloride

Into a suitable 3-neck flask equipped with a thermometer, motorized stirrer, and addition funnel, place a sodium hydroxide solution prepared by adding and dissolving 11.4 grams of sodium hydroxide into 114 milliliters of cold water. Note: sodium hydroxide generates much heat when dissolved in water, so allow the solution to cool to room temperature before using. Then place this 3-neck flask into a cold-water bath, and chill to about 20 Celsius. When the temperature of the sodium hydroxide solution reaches about 20 Celsius, place 12.5 grams of liquid bromine into the addition funnel, and then slowly add it, drop-wise, to the sodium hydroxide solution of period of time sufficient to keep the sodium hydroxide solution below 25 Celsius. During the addition of the liquid bromine, rapidly stir the sodium hydroxide solution. After the addition of the liquid bromine, remove the addition funnel (just used), and replace it with a conventional funnel. Thereafter, immediately add in 13.7 grams of the product obtained in step 5 (note: make sure the product obtained in step 5 is finely ground into a powder before adding). The rate of addition of the 13.7 grams of product obtained in step 5 should be rather fast. Note: during the addition, rapidly stir the reaction mixture, and maintain its temperature below 25 Celsius. After the addition of the product obtained in step 5 (make sure it all dissolves into the reaction mixture), heat the entire reaction mixture at 70 Celsius for about 15 minutes. During the heating process, continue to stir the reaction mixture. After 15 minutes, add in (generally fast) 12.5 grams of solid sodium hydroxide, and then raise the temperature of the reaction mixture to 80 Celsius shortly thereafter. Then heat the reaction mixture at 80 Celsius with constant stirring for about 30 minutes. After heating for 30 minutes, remove the heat source, and allow the reaction mixture to cool to room temperature. Thereafter, extract the entire reaction mixture with three 75-milliliter portions of diethyl ether. After the extraction process, combine all ether portions, if not already done so, and then add to this combined ether mixture 15 grams of anhydrous magnesium sulfate (to absorb water), and then stir the entire ether mixture for 10 minutes. Then simply filter-off the magnesium sulfate. Note: at this point, the filtered ether mixture will contain the freebase β-ortho-methoxy phenyl-n-propylamine. Finally, place the filtered ether mixture into a clean beaker, and then place this beaker into an ice bath, and chill to about 10 Celsius. Thereafter, bubble into the ether mixture, 2 grams of dry hydrogen chloride gas. After the addition of the hydrogen chloride gas, filter-off any precipitated product, and then vacuum dry or air-dry this filtered-

off product. Note: additional product can be obtained by removing the ether either by a distillation apparatus, or by a rotary evaporator until dry solid remains. This dry solid can then be combined with the previously dried collected product.

Note: Other salts of the freebase β-ortho-methoxy phenyl-n-propylamine such as sulfate, tartrate, citrate, and phosphate can be prepared by adding the corresponding acid to the ether mixture obtained at the end of step 6. For sulfuric acid or tartaric acid, 2.3 grams of sulfuric acid or 3.5 grams of d-tartaric acid should be added to the ether mixture; and for citric acid or phosphoric acid, 3 grams of citric acid or 1.5 grams of phosphoric acid should be added to the ether mixture. For each case, the ether mixture should then be filtered to recover the desired salt, and the resulting desired salt then vacuum dried or air-dried. The ether mixture in each of these cases can be evaporated using a distillation apparatus, or rotary evaporator only to the point where 80% of the total volume is reduced. The resulting ether concentrate can then be filtered to recover any additional product, which can then be vacuum dried or air-dried and then combined with the previously collected product. All the salts are stimulants with similar effects as the hydrochloride.

0006-02. β-o-Methoxyphenyl propylmethylamine hydrochloride. *1-methoxy-2-(1-methylbutyl)benzene hydrochloride*



Structure	3D model

β-ortho-methoxy phenyl-n-propylamine forms colorless to whitish, to brownish crystals (depending on purity) with a melting point of 199 Celsius. The crystals are more potent then the corresponding β-ortho-methoxy phenyl-n-propylamine hydrochloride or other salts thereof, and is effective for treating symptoms of fatigue, and depression. The crystals can also be used to invoke feelings of stimulation and well-being when sniffed or inhaled. Intravenous administration results in extra ordinary feelings of stimulation, energy bursts, and feeling of well-being—especially when admixed with a mild pain killer. Note: This compound has been used in the treatment of asthma, and nasal congestion.

Note: This substance is a controlled substance (stimulant) as listed in the US code of Federal regulations.

Toxicity: Moderate	**Rate of onset (average):** Moderately low
Stimulation dosage (ingestion): 35 to 45 milligrams	**Duration of stimulation (average):** 5 to 7 hours (depending on the person)
Stimulation dosage (inhalation): 25 to 30 milligrams	**Habit forming potential:** Moderate
Stimulation dosage (injection): 10 milligrams +	**Estimated value U.S. (based on procedure):** $18 per gram

Procedure A: Preparation of β-o-Methoxyphenyl propylmethylamine hydrochloride

Materials:

1. 16.5 grams of freebase β-ortho-methoxy phenyl-n-propylamine (procedure 0006, step 6)	6. 10 milliliters of glacial acetic acid
2. 10.6 grams of benzaldehyde	7. 275 milliliters of diethyl ether
3. 50 milliliters of 95% ethyl alcohol	8. 150 milliliters of a 33% potassium hydroxide solution
4. 9.4 grams of methyl iodide	9. 15 grams of anhydrous magnesium sulfate
5. 66 milliliters of methyl alcohol (methanol)	10. 4 grams of dry hydrogen chloride gas

Summary: β-o-Methoxyphenyl propylmethylamine hydrochloride is prepared in simple two-step process starting with the formation of β-ortho-methoxyphenylbenzalamine. This product is prepared by reacting the freebase of β-ortho-methoxy phenyl-n-propylamine (prepared in process 0006 at the end of step 6) with benzaldehyde I the presence of ethyl alcohol under reflux. The resulting β-ortho-methoxyphenylbenzalamine is then recovered, and then converted into the β-o-Methoxyphenyl propylmethylamine freebase by reaction with methyl iodide under heat and pressure. The resulting reaction mixture is then hydrolyzed with methyl alcohol in the presence of water; the resulting mixture is then distilled, and then acidified with acetic

acid. The acidic mixture is then basified by the addition of potassium hydroxide, and the resulting mixture is then extracted with ether to recover the freebase of β-o-Methoxyphenyl propylmethylamine, which is then converted to the hydrochloride by the addition of hydrogen chloride gas. For related information, see serial number: 386,661, April 3rd, 1941 by Eugene H. Woodruff, from Kalamazo, Mich., to The UpJohn Company.

Hazards: Extinguish all flames before using diethyl ether, ethyl alcohol, methyl alcohol, and glacial acetic acid. Ether can form explosive mixtures with air. Wear gloves when handling glacial acetic acid, potassium hydroxide, and methyl iodide, all of which can cause irritation. Note: keep methyl iodide stored in amber glass bottles away fro sunlight.

Procedure:

Personnel notes for procedure A: β-o-Methoxyphenyl propylmethylamine hydrochloride

Step 1: Preparation of β-ortho-methoxyphenylbenzalamine

To the ether mixture of the freebase β-ortho-methoxy phenyl-n-propylamine (obtained at the end of step 6—procedure 0006), place this ether mixture into a distillation apparatus, or rotary evaporator, and remove the ether. When no more ether is collected, remove the remaining left over oily liquid (which will the freebase β-ortho-methoxy phenyl-n-propylamine). Now, place 16.5 grams of this freebase compound into suitable reflux apparatus, and then add in 10.6 grams of benzaldehyde, followed by 25 milliliters of 95% ethyl alcohol. Then reflux this mixture at 78 Celsius for about 15 minutes. After the reflux period, quickly replace the reflux condenser with a conventional condenser (fitted with a receiver flask), and then boil-off the ethyl alcohol and the water by heating and distilling at 100 Celsius until no more water or alcohol is collected. When no more water or alcohol can be removed remove the remaining oily residue (after it has cooled to room temperature), and then set aside for step 2. Note: this collected product can then be vacuum distilled at 185 celsius under a vacuum of 10 millimeters of mercury to obtain a refined product, but this vacuum distillation process is not needed for step 2. **Note: The collected product will be the β-ortho-methoxyphenylbenzalamine, which can be used as a mild stimulant if desired.**

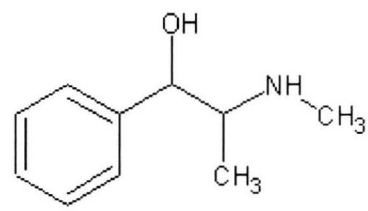
Step 2: Preparation of β-o-Methoxyphenyl propylmethylamine hydrochloride

Into a suitable standard flask, place 16. 9 grams of the product obtained in step 1, and then add in 9.4 grams of methyl iodide. Now, place a large, tightly fitting balloon over the flask, and then heat the contents in this flask to about 100 Celsius for 40 minutes. Note: The balloon is placed over the flask not only to create an airtight seal, but to create pressure as the contents are heated. When the contents in the flask are heated, the pressure will cause the balloon to inflate. In most cases, a metal circular screw clamp should be used to securely fasten the balloon to the flask. During the heating process, the balloon will inflate and expand, but should not blow. This inflation and expansion will produce enough pressure inside the flask to properly carryout the reaction of the methyl iodide. If the balloon does happen to blow or break, quickly replace it with another one, and continue the heating process for the remaining amount of time. After heating the contents in the flask in this manner for 40 minutes, remove the heat source and allow the contents in the flask to cool to room temperature. Note: during this cool down, watch the balloon so it does not get sucked into the flask due to backpressure. When the contents have cooled to room temperature, remove the balloon, and then place the entire contents of the flask into a beaker. Thereafter, add to the contents in this beaker, 66 milliliters of methanol, followed by 66 milliliters of water. Then place this entire mixture into suitable sized reflux apparatus, and then reflux this mixture at 68 Celsius for about 15 minutes. After refluxing for 15 minutes, quickly replace the reflux condenser with a conventional condenser (fitted with a receiver flask), and then distill-off the methanol and any left over benzaldehyde by heating and distilling at 100 Celsius. Note: to aid in the removal of any benzaldehyde, add to the reflux apparatus just before starting the distillation, 100 milliliters of hot water (to aid in steam distillation of the benzaldehyde). After distilling at 100 Celsius for about 1 hour, remove the heat source, and allow the left over remaining contents in the apparatus to cool to room temperature. Then remove said contents and then place the contents into a clean beaker. Then add in 10 milliliters of glacial acetic acid, and then stir the entire mixture for about 15 minutes. Thereafter, quickly extract the entire mixture with two 25-milliliter portions of diethyl ether (to remove any coloring matter or impurities), and then discard or recycle these two ether portions thereafter. Then, add to the just extracted mixture, 150 milliliters of a 33% potassium hydroxide solution, and then stir the entire mixture for about 15 minutes. Now, extract this basic mixture with three 75-millilier portions of diethyl ether (to recover the freebase product). After the extraction process, combine all ether extracts (if not already done so), and then dry this combined ether portions by adding to it, 15 grams of anhydrous magnesium sulfate (to absorb water), and then briefly stir the mixture for about 10 minutes—thereafter, filter-off the magnesium sulfate. The filtered ether mixture will

contain the freebase of β-o-Methoxyphenyl propylmethylamine. Now to this ether mixture containing the freebase product, add in about 25 milliliters of 95% ethyl alcohol, and then place this mixture into an ice bath, and chill to about 10 Celsius. Then bubble into this mixture 4 grams of dry hydrogen chloride gas. Note: during the addition, stir the mixture. After the addition of the hydrogen chloride, stir the entire mixture for 1 hour at 10 Celsius. Then filter-off any precipitated product, and then vacuum dry or air-dry this filtered-off product. Note: some additional product can be obtained by evaporating-off the ether and ethyl alcohol using a distillation apparatus until only 80% of the total volume has been reduced. Any additional product can then be filtered-off, and then vacuum dried or air-dried in the usual manner, and then combined with any previous collected product. **Note:** the pressure process discussed above, whereby a balloon is placed over a flask, can be substituted by using a conventional steel pipe with threads at both ends. To carryout the steel pipe technique, pour all necessary materials (as describe for the balloon technique), into a thick walled stainless steel pipe, and then seal both ends with the corresponding steel caps. The threads at each end should be wrapped with Teflon tape prior to screwing in the end caps. Then place the entire pipe, and submerge it into a water bath and heat at the desired temperature for the desired time. Note: this process can be dangerous and can lead to pressure explosions. Carryout the process in an area that can contain any such explosion, and maintain a safe distance away during the operation—just to be on the safe side.

Note: Other salts of the freebase β-o-Methoxyphenyl propylmethylamine such as sulfate, tartrate, citrate, and phosphate can be prepared by adding the corresponding acid to the ether mixture obtained at the end of step 2. For sulfuric acid or tartaric acid, 3 grams of sulfuric acid or 3.9 grams of d-tartaric acid should be added to the ether mixture; and for citric acid or phosphoric acid, 3.5 grams of citric acid or 2.5 grams of phosphoric acid should be added to the ether mixture. For each case, the ether mixture should then be filtered to recover the desired salt, and the resulting desired salt then vacuum dried or air-dried. The ether mixture in each of these cases can be evaporated using a distillation apparatus, or rotary evaporator only to the point where 80% of the total volume is reduced. The resulting ether concentrate can then be filtered to recover any additional product, which can then be vacuum dried or air-dried and then combined with the previously collected product. All the salts are stimulants with similar effects as the hydrochloride.

Intermediate-0007. Ephedrine. *2-(methylamino)-1-phenylpropan-1-ol*



Ephedrine is an interesting compound that has several forms. The DL-form forms colorless to whitish crystals with a melting point of 79 Celsius. The L-form forms colorless to white crystals with a melting point of 34 Celsius, and the crystals may contain up to ½ molecule of water of hydration. Ephedrine also forms a psudoephedrine with a melting point of 119 Celsius. Ephedrine is natural occurring, and exists in the aforementioned forms from Chinese Ma Huang herb (Ephedra vulgaris, E. sinica Stapf., E. equisetina Bunge, Gnetaceae), from which it can be extracted with several solvents. Ephedrine is widely used in cold and nasal products and for use as a bronchodilator (the L-form). It is widely used in the preparation of methamphetamine when reduced with several reagents including hydroiodic acid/phosphorus, sodium/mercury amalgam, or by sodium or lithium reduction in suitable media. Note: Ephedrine is not a controlled substance, but pressure by anti-drug groups on legislation may have lead to new regulations. Check your local listings for potential regulations in regards to this substance. It will be concluded that this substance most likely will become more regulated due to its role in the preparation of methamphetamines.

Procedure A: Preparation of ephedrine (DL and L forms)

Materials:

1. 53 grams of benzaldehyde	7. 7 grams of a 37% formaldehyde solution
2. 37 grams of nitroethane	8. 6 grams of finely divided zinc
3. 30 milliliters of a 30% potassium carbonate solution	9. 6 grams of glacial acetic acid
4. 650 milliliters of diethyl ether	10. 6 grams of hydrogen sulfide gas
5. 90 milliliters of a 10% sodium bisulfite solution	11. 10 milliliters of 35 to 38% hydrochloric acid
6. 30 grams of anhydrous magnesium sulfate	12. 6 grams of anhydrous sodium carbonate

Summary: Ephedrine is a major starting material for the preparation of methamphetamine and other stimulants. In this process, ephedrine is prepared by the condensation of benzaldehyde with nitroethane in the presence of potassium carbonate. The reaction is rather general, and afterwards, the mixture is treated with diethyl ether, and then a solution of sodium bisulfite, which removes any unreacted benzaldehyde. Thereafter, the ether layer is recovered, washed, and then dried, and the resulting ether mixture then evaporated to recover the intermediate product, phenylnitropropanol. This phenylnitorpropanol is then reduced by reacting it with formaldehyde in the presence of acetic acid and zinc dust. After the reduction, the reaction mixture is treated with hydrogen sulfide (to precipitate zinc), and the resulting mixture is then filtered, and then treated with ether followed by hydrochloric acid. The ether layer is then removed, and the lower water layer is then treated with sodium carbonate to liberate the freebase of ephedrine. The freebase is then extracted into ether, the ether layer is then recovered, and then evaporated to recover the desired ephedrine. For related information, see Serial number, 88,224, April 1st, 1916 by Wilhelm Nagajoshi, of Tokyo Japan, to M. Dick Bunnell, of San Francisco, CA; also see Serial number, 433,816, March 6th, 1930, by Chogi Nagai of Tokyo, Japan, to Alexander Nagai, of Berlin Germany.

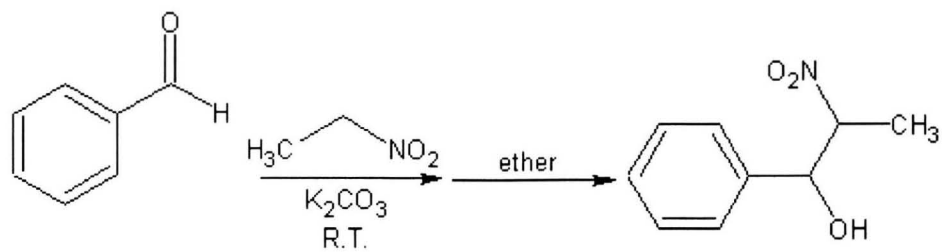
Hazards: Use care when handling nitroethane, and diethyl ether, both of which form explosive mixtures with air and are highly flammable. Use caution when handling hydrogen sulfide gas, which is very toxic and flammable. Use maximum ventilation when handling concentrated formaldehyde solution, and avoid inhalation. Wear gloves when handling concentrated acetic acid, and hydrochloric acid, as they are both corrosive and can cause irritation.

Procedure:

Personnel notes for procedure A: Ephedrine

Step 1: Preparation of phenylnitropropanol

Into a suitable beaker or flask, place 53 grams of benzaldehyde, followed by 37 grams of nitroethane. Immediately thereafter, add in 30 milliliters of a 30% potassium carbonate solution, and rapidly stir the entire mixture at room temperature for 2 hours. Note: A cold-water bath may or may not be needed to keep the reaction mixture at ambient temperature (room temperature). Do not allow the reaction mixture to get above 25 Celsius. After stirring for 2 hours, add to the reaction mixture 200 milliliters of diethyl ether, and shortly thereafter, add in 90 milliliters of a 10% sodium bisulfite solution, and then moderately stir the entire reaction mixture for 30 minutes. Afterwards, place the entire reaction mixture into a seperatory funnel, and then remove the upper ether layer (after removing the lower aqueous layer). Thereafter, wash this upper ether layer with three 75-milliliter portions of cold water. Note: after each washing portion, use a seperatory funnel to recover the ether layer, which will be the upper layer each time. After the washing portion, add to the ether layer, 15 grams of anhydrous magnesium sulfate (to absorb water), and then stir the entire mixture for 10 minutes. Then filter-off the magnesium sulfate. Then, place the filtered ether mixture into a distillation apparatus, or rotary evaporator, and remove the ether. When no more ether passes over or is collected, remove the remaining oily residue (after allowing it to cool to room temperature), and then place aside for step 2. This oily residue will consist of the desired phenylnitropropanol.

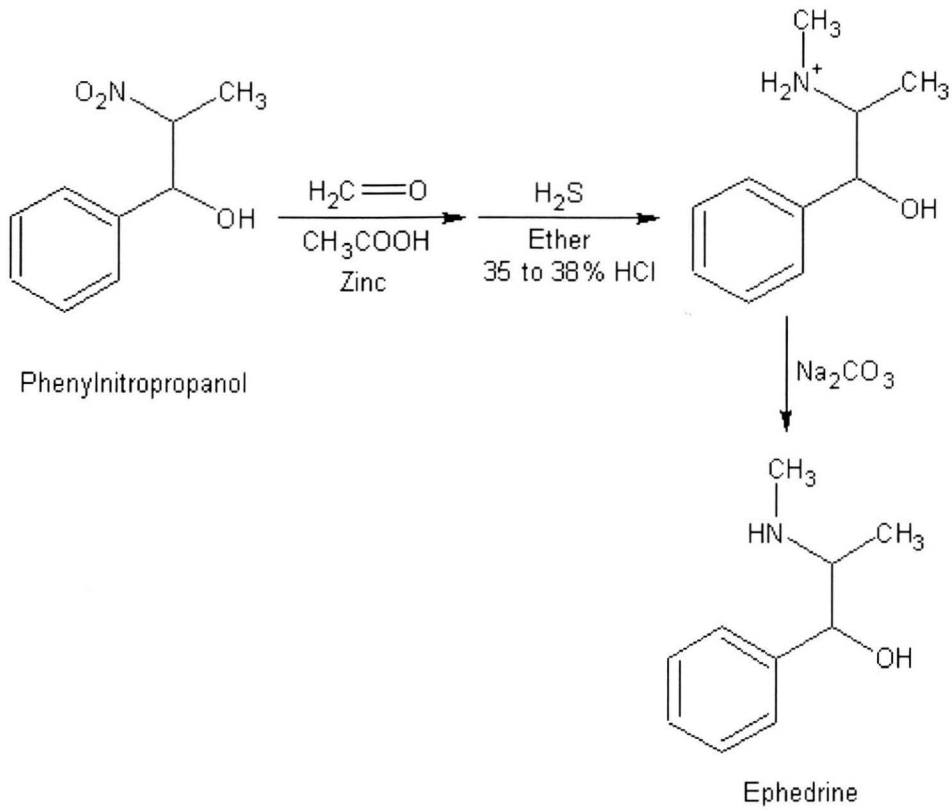


Benzaldehyde

Phenylnitropropane

Step 2: Into a suitable flask equipped with motorized stirrer, and gas inlet tube, place 15 grams of the oily residue obtained in step 1, and then place this flask into an ice bath, and chill to 0 Celsius. When the oily residue from step 1 reaches a temperature of about 0 Celsius, add in 7 grams of a 37% formaldehyde solution. Thereafter, begin to stir these contents vigorously, and then carefully add in an acetic acid solution (prepared by adding and dissolving 6 grams of glacial acetic acid into 14 milliliters of ice cold water), and then carefully add in 6 grams of finely divide zinc (preferable zinc dust). During the addition of the acetic acid solution and zinc, vigorously stir the reaction mixture, and maintain its temperature below 5 celsius at all times. After the

addition of the zinc, continue to stir the reaction mixture below 5 Celsius for about 90 minutes to complete the reaction. After 90 minutes, stop stirring, and then filter-off any insoluble zinc or other materials. Thereafter, place the filtered reaction mixture back into the ice bath, and then bubble into this filtered reaction mixture, 6 grams of hydrogen sulfide gas. Note: during the addition, vigorously stir the reaction mixture. Note: the addition of the hydrogen sulfide will precipitate any dissolved zinc. After the addition of the hydrogen sulfide, filter-off any insoluble materials, and then add to this filtered mixture, 250 milliliters of cold water, followed by 150 milliliters of diethyl ether, followed by 10 milliliters of a 35 to 38% hydrochloric acid solution. Then vigorously stir this entire mixture for about 1 hour. After 1 hour, place the entire reaction mixture into a seperatory funnel, and remove the lower water layer (which will contain the desired ephedrine product as the hydrochloride). Note: the upper ether layer can be discarded or recycled if desired. Now, to the recovered lower water layer, add in a sodium carbonate solution prepared by adding and dissolving 6 grams of anhydrous sodium carbonate into 15 milliliters of cold water, and then stir the whole mixture for about 15 minutes. Finally, extract this entire mixture with three 100-milliliter portions of diethyl ether, and after the extraction process (after each extraction, the ether will be the upper layer), combine all ether portions, if not already done so, and then dry this combined ether portion by adding in 15 grams of anhydrous magnesium sulfate. Then stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Thereafter, place this filtered ether mixture into a distillation apparatus or rotary evaporator, and remove the ether. When no more ether is recovered, recover the left over remaining residue (after it has cooled to room temperature), and then store in an appropriate amber glass bottle until use.



Phenylnitropropanol

Ephedrine

Note: this freebase ephedrine will actually be a mixture of the DL and L-forms, from which the L-form is the most common used in the preparation of methamphetamine. However, regardless of other literature, methamphetamines can be prepared using either the DL-form or the L-form with satisfactory results.

Note: Other salts of the freebase ephedrine such as the sulfate, tartrate, citrate, and phosphate can be prepared by adding the corresponding acid to the freebase compound obtained at the end of step 2 (the freebase should be dissolved into ether in the ratio of 1 grams of freebase to 15 grams of diethyl ether). For the hydrochloride, 1 mole of acid gas should be bubbled into the ether mixture for every 1 mole of freebase ephedrine (not for the total weight of the ether mixture). For sulfuric acid or tartaric acid, 1 mole of sulfuric acid or d-tartaric acid should be added for 2 moles of the freebase ephedrine (not for the total weight of the ether mixture); and for citric acid or phosphoric acid, 1 mole of the citric acid or phosphoric acid should be added to 3 moles of the freebase ephedrine (not for the total weight of the ether mixture). The ether mixture in each of these cases can be evaporated using a distillation apparatus, or rotary evaporator only to the point where 80% of the total volume is reduced. The resulting ether concentrate can then be filtered to recover the product, which can then be vacuum dried or air-dried. All the salts of ephedrine are mild stimulants, but can be used in the preparation of stimulant compositions (by addition to amphetamines, or other CNS stimulants), or can be used in the preparation of other stimulants.

Procedure B: Preparation of ephedrine (predominately the DL form)

Materials:

1. 107 grams of benzaldehyde	8. 6 grams of finely divided zinc
2. 82.5 grams of nitroethane	9. 6 grams of glacial acetic acid
3. 45 grams of sodium hydroxide	10. 6 grams of hydrogen sulfide gas
4. 650 milliliters of diethyl ether	11. 10 milliliters of 35 to 38% hydrochloric acid
5. 210 grams of sodium bisulfite	12. 6 grams of anhydrous sodium carbonate
6. 35 grams of anhydrous magnesium sulfate	13. 25 milliliters of 95% ethyl alcohol
7. 7 grams of a 37% formaldehyde solution	

Summary: DL-ephedrine is prepared in an identical manner as for procedure A, but a mother liquor of benzaldehyde and sodium bisulfite is utilized in combination with a caustic solution of sodium hydroxide and nitroethane. The reaction is rather general, and the desired phenylnitropropanol is recovered in the usual manner. Step 2 involves the usual reduction process for reducing the phenylnitropropanol to DL-ephedrine.

Hazards: Use care when handling nitroethane, and diethyl ether, both of which form explosive mixtures with air and are highly flammable. Use caution when handling hydrogen sulfide gas, which is very toxic and flammable. Use maximum ventilation when handling concentrated formaldehyde solution, and avoid inhalation. Wear gloves when handling concentrated acetic acid, and hydrochloric acid, as they are both corrosive and can cause irritation.

Procedure:

Personnel notes for procedure B: Ephedrine

Step 1: Preparation of phenylnitropropanol

Into a suitable flask equipped with motorized stirrer, place 107 grams of technical grade benzaldehyde, and immediately thereafter, pour in a sodium bisulfite solution prepared by adding and dissolving 110 grams of sodium bisulfite into 500 milliliters of water. Thereafter, vigorously stir the entire mixture at ambient temperature (room temperature) for about 3 hours. Then, prepare a solution by adding 45 grams of sodium hydroxide into 200 milliliters of water, and then after this caustic solution cools to room temperature, add in 82.5 grams of nitroethane. Note: sodium hydroxide generates much heat when dissolved in water, so allow the solution to cool to room temperature before using. Once the nitroethane/caustic solution has been prepared, gently and briefly warm it to about 30 Celsius, and when its temperature reaches 30 Celsius, pour the entire solution into the benzaldehyde/sodium bisulfite reaction mixture, and then vigorously stir the entire reaction mixture for about 30 minutes. After 30 minutes, stop stirring, and allow the entire reaction mixture to stand overnight. The next day, pour the entire reaction mixture into a seperatory funnel (note: insoluble solids may deposit overnight, if this is the case, simply filter-off any insoluble solids before pouring the reaction mixture into a seperatory funnel), and then remove the lower water layer (the desired upper layer will be referred to as the organic layer). Note: in some cases, the water layer may be the upper layer. After removal of the water layer, discard it, or recycle it if desired, and then place the organic layer into a suitable beaker, and then add in a fresh solution of sodium bisulfite prepared by adding 110 grams of sodium bisulfite into 500 milliliters of water, and then vigorously stir the entire mixture for about 15 minutes at ambient temperature. After stirring for 15 minutes, stop string and then allow the entire mixture to stand overnight once again. The following day, filter-the reaction mixture to remove any insoluble solids (not layers), and then place the filtered mixture into a seperatory funnel, and then remove the upper organic layer (desired layer), after removing the lower water layer first. Note: in some cases the desired organic layer may be the lower layer. [Second note: the water layer, or aqueous layer, can be recycled rather then discarded. To do this, it should be added to the previously discarded water layer (aqueous layer), and then treated with another 107-gram portion of benzaldehyde, and the process started over again as indicated in the start of this entire procedure]. The recovered organic layer, can then be dried by adding to it, 20 grams of anhydrous magnesium sulfate (to absorb water), and stirred briefly for about 10 minutes at room temperature—thereafter, filter-off the magnesium sulfate. The filtered organic layer, which is the desired phenylnitropropanol, can then be set aside for step 2. If desired, it can be vacuum distilled at 165 Celsius under vacuum of 5 millimeters of mercury to obtain a colorless, odorless oily liquid of high purity, but this is not generally needed for step 2. Note: if desired, the phenylnitropropanol can be extracted with diethyl ether or methylene chloride (ratio of 1 gram product to 5 grams solvent), the resulting mixture filtered, and then evaporated to yield a refined phenylnitropropanol.

Step 2: Preparation of DL-ephedrine

Into a suitable flask equipped with motorized stirrer, and gas inlet tube, place 15 grams of the oily organic product obtained in step 1, followed by a dilute alcohol solution prepared by mixing 25 milliliters of 95% ethyl alcohol into 120 milliliters of ice cold water, and then stir this entire mixture to dissolve the oily organic product from step 1. Thereafter, place this flask into an ice bath, and chill to 0 Celsius. When the contents of the flask reach a temperature of about 0 Celsius, add in 7 grams of a 37% formaldehyde solution. Thereafter, begin to stir these contents vigorously, and then carefully add in an acetic acid solution (prepared by adding and dissolving 6 grams of glacial acetic acid into 14 milliliters of ice cold water), and then carefully add in 6 grams of finely divide zinc (preferable zinc dust). During the addition of the acetic acid solution and zinc, vigorously stir the reaction mixture, and maintain its temperature below 5 celsius at all times. After the addition of the zinc, continue to stir the reaction mixture below 5 Celsius for about 90 minutes to complete the reaction. After 90 minutes, stop stirring, and then filter-off any insoluble zinc or other materials. Thereafter, place the filtered reaction mixture back into the ice bath, and then bubble into this filtered reaction mixture, 6 grams of hydrogen sulfide gas. Note: during the addition, vigorously stir the reaction mixture. Note: the addition of the hydrogen sulfide will precipitate any dissolved zinc. After the addition of the hydrogen sulfide, filter-off any insoluble materials, and then add to this filtered mixture, 250 milliliters of cold water, followed by 150 milliliters of diethyl ether, followed by 10 milliliters of a 35 to 38% hydrochloric acid solution. Then vigorously stir this entire mixture for about 1 hour. After 1 hour, place the entire reaction mixture into a seperatory funnel, and remove the lower water layer (which will contain the desired DL-ephedrine product as the hydrochloride). Note: the upper ether layer can be discarded or recycled if desired. Now, to the recovered lower water layer, add in a sodium carbonate solution prepared by adding and dissolving 6 grams of anhydrous sodium carbonate into 15 milliliters of cold water, and then stir the whole mixture for about 15 minutes. Finally, extract this entire mixture with three 100-milliliter portions of diethyl ether, and after the extraction process (after each extraction, the ether will be the upper layer), combine all ether portions, if not already done so, and then dry this combined ether portion by adding in 15 grams of anhydrous magnesium sulfate. Then stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Thereafter, place this filtered ether mixture into a distillation apparatus or rotary evaporator, and remove the ether. When no more ether is recovered, recover the left over remaining residue (after it has cooled to room temperature), and then store in an appropriate amber glass bottle until use.

Note: Other salts of the freebase DL-ephedrine such as the sulfate, tartrate, citrate, and phosphate can be prepared by adding the corresponding acid to the freebase compound obtained at the end of step 2 (the freebase should be dissolved into ether in the ratio of 1 grams of freebase to 15 grams of diethyl ether). For the hydrochloride, 1 mole of acid gas should be bubbled into the ether mixture for every 1 mole of freebase DL-ephedrine (not for the total weight of the ether mixture). For sulfuric acid or tartaric acid, 1 mole of sulfuric acid or d-tartaric acid should be added for 2 moles of the freebase DL-ephedrine (not for the total weight of the ether mixture); and for citric acid or phosphoric acid, 1 mole of the citric acid or phosphoric acid should be added to 3 moles of the freebase DL-ephedrine (not for the total weight of the ether mixture). The ether mixture in each of these cases can be evaporated using a distillation apparatus, or rotary evaporator only to the point where 80% of the total volume is reduced. The resulting ether concentrate can then be filtered to recover the product, which can then be vacuum dried or air-dried. All the salts of DL-ephedrine are mild stimulants, but can be used in the preparation of stimulant compositions (by addition to amphetamines, or other CNS stimulants), or can be used in the preparation of other stimulants.

Intermediate-0007-02. Extraction of L-ephedrine from Ma Huang herb

Materials:

1. 200 grams of ma huang herb	5. 225 milliliters of 10% hydrochloric acid solution
2. 450 milliliters of diethyl ether	6. 300 milliliters of 10% sodium hydroxide solution
3. 100 milliliters of 10% ammonia solution	7. 200 grams of sodium chloride
4. 110 grams of anhydrous sodium carbonate	8. 20 grams of dry hydrogen chloride gas

Hazards: Extinguish all flames before using diethyl ether, which is highly flammable, and can form explosive mixtures with air. Wear gloves and use proper ventilation when handling ammonia water, which is very irritating—avoid prolonged inhalation of the vapors. Wear gloves when handling sodium hydroxide and hydrochloric acid, both of which are capable of causing skin irritation.

Process:

Personnel notes for intermediae-0007-02: L-ephedrine

Into a standard lab blender (just like a standard kitchen blender with stainless steel blade), place 200 grams of ma huang herb (obtained from a number of sources such as herbal stores, on-line sites, ect., ect.,), and then add in 450 milliliters of diethyl ether. Thereafter, blend the entire mixture at slow speed (note: a quick rapid blend may be needed in order to mutilate or chop up the ma huang herb for proper blending) for about 1 hour. After 1 hour, add in 100 milliliters of a 10% ammonia solution, followed by 10 grams of anhydrous sodium carbonate, and then blend the entire mixture for about 6 hours at a slow speed setting. After blending the mixture for 6 hours, stop the blending, and then allow the entire mixture to stand overnight. The next day, slowly begin the stiring process again, and then add in three 75-milliliter portions of 10% hydrochloric solution, followed thereby with three 100-milliliter portions of 10% sodium hydroxide solution. After all additions, slowly blend the entire mixture for 1 hour. After 1 hour, add in 100 grams of anhydrous sodium carbonate, followed by 200 grams of sodium chloride to saturate the entire mixture. After the addition of the salt, slowly blend the entire mixture for about 30 minutes. After 30 minutes, stop the blending procedure, and then filter the entire mixture to remove insoluble matter, and then remove the upper ether layer from the filtered mixture by using a seperatory funnel or by decantation. Once the ether layer has been collected, place it into a distillation apparatus, and gently remove the ether until only 70% of the total volume remains. Once this point is reached, stop the distillation process, and allow the remaining ether concentrate to cool to room temperature before removing it from the apparatus. Thereafter, quickly filter this ether concentrate to remove any insoluble materials, and then place this filtered ether concentrate into a clean beaker, and then place this beaker into an ice bath, and chill to 0 Celsius. Thereafter, bubble into this chilled ether mixture, 20 grams of dry hydrogen chloride gas (to the point of saturation). Afterwards, filter-off any precipitated products, and then vacuum dry or air-dry the collected precipitates. The dry solids obtained will consist of predominately L-ephedrine, with small amounts of D-pseudoephedrine, and small amounts of impurities.

Note: obviously there are numerous modifications to this process, and the person carrying out this process is encouraged to experiment and try different solvents and techniques.

Intermediate-0007-03. Extraction of pseudoephedrine from store bought pseudoephedrine tablets ("Sudafed" "Galpseud", "Novafed", "Rhinalair", "Otrinol", "Sinufed", Symptom 2", "Afrinol", and other nasal decongestants and/or bronchodilators)

Materials:

1. 1.5 grams worth of store bought pseudoephedrine pills	7. 5 to 10 milliliters of 99% isopropyl alcohol
2. 50 milliliters of turpentine	8. 30 milliliters of toluene or xylene
3. 50 milliliters of toluene	9. 5 grams of anhydrous sodium sulfate
4. 75 milliliters of acetone	10. 70 milliliters of dry diethyl ether
5. 2 grams of anhydrous sodium carbonate	11. 5 grams of dry hydrogen chloride gas
6. 2 grams of sodium chloride	

Hazards: Extinguish all flames before using diethyl ether, which is highly flammable, and can form explosive mixtures with air. Follow similar guidelines when handling acetone, which is also highly flammable. Inhalation of toluene or xylene vapors should be avoided, as toluene and xylene are listed as suspected carcinogens. Wear gloves when handling sodium hydroxide and hydrogen chloride gas, both of which are capable of causing skin irritation.

Procedure:

Personnel notes for intermediate-0007-03: Pseudoephedrine

Note: the below process (process 1A) works for all generic brands of pseudoephedrine, and the so called "time" release tablets that may contain up to 120 to 240 milligrams worth of pseudoephedrine per tablet. Many brands of pseudoephedrine, other then just the generic brands can be used with success. The following lists of ingredients are commonly found in both the generic and name brand products, and can be used with satisfactory results.

Formulation 1

I. Main ingredients	Pseudoephedrine hydrochloride: 60 milligrams Tripolidine: 2.5 milligrams
II. Inert ingredients	Corn starch, flavor, hydroxypropyl methylcellulose, lactose, magnesium stearate, polyethylene glycol, potato starch, povidone, sucrose, and titanium dioxide

Formulation 2

I. Main ingredients	Pseudoephedrine hydrochloride: 60 milligrams Tripolidine: 2.5 milligrams
II. Inert ingredients	Starch, flavor, hydroxypropyl methylcellulose, magnesium stearate, polyethylene glycol, titanium dioxide, with one or more of the following: cellulose, dioctyl sodium sulfosuccinate, hydroxypropyl cellulose, lactose, polysorbate 80, povidone, powdered cellulose, pregelatinized starch, silica gel, silicon dioxide, sodium starch glycolate, and/or stearic acid

Formulation 3

I. Main ingredients	Pseudoephedrine hydrochloride: 60 milligrams Tripolidine: 2.5 milligrams
II. Inert ingredients	Corn starch, hydroxypropyl methylcellulose, flavor, lactose, carnuba wax, magnesium stearate, polyethylene glycol, povidone, and sucrose

Formulation 4 – timed release brand

I. Main ingredients	Pseudoephedrine hydrochloride: 120 or 240 milligrams
II. Inert ingredients	Candellia wax, hydroxypropyl methylcellulose, magnesium stearate, microcrystalline cellulose, polyethylene glycol, povidone, titanium dioxide, or Candellia wax, hydroxypropyl methylcellulose, magnesium steraate, microcrystalline cellulose, polyethylene glycol, povidone, titanium dioxide

Process 1A: Preparation of freebase pseudoephedrine

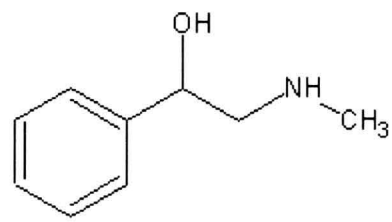
Some store bought pseudoephedrine tablets have a red coating to them. These red coatings are distinct in nature to the specified manufacture. To extract the pseudoephedrine from these red-coated tablets (30 to 60 milligram tablets), gently scrape-off the red coating before procedure. Other pseudoephedrine tablets may contain other colored coatings or similar natured shells. If this is the case, the outer coating in all cases should be gently scraped-off under the usual means, before proceeding. Thereafter, in either case, take 1.5 grams worth of scraped pills or similar natured pills (25+ tablets), and ground them into a fine powder. Note: it does not matter whether the pseudoephedrine in the store bought tablets is the hydrochloride, sulfate, maleate, citrate, phosphate, or tartrate. Now, we must gently blend this ground powder with a suitable solvent, but which solvent we use is determined by the specific "inert" ingredient within the tablets. If one of the inert ingredients in the original tablets is a compound called "povidone", place the powdered tablets into 50 milliliters of turpentine, and then gently stir the entire mixture at room temperature for about 11 to 12 hours. If an inert ingredient in the original tablets is "polyethylene glycol", rather then the povidone, place the powdered tablets into 50 milliliters of toluene, and then gently stir this entire mixture at room temperature for about 5 to 6 hours. **Note: some brands of store bought pseudoephedrine contain both povidone and polyethylene glycol, if this is the case, use the turpentine stirring technique.** After gently stirring for the necessary amount of time (either way), filter-off the insoluble solids, and then vacuum dry or air-dry them. The solvent used for the gentle stirring can be recycled or discarded if desired. Now, if the original tablets did not contain any antihistamine compound, wash the dried filtered-off solids with two 25-millilter portions of dry acetone (several times using the same washing portions), and then vacuum dry or air-dry the washed solids. If however, the original tablets contained an antihistamine compound, place the dried filtered-off solids into a suitable sized beaker, and then add in 25 milliliters of dry acetone. Thereafter, gently heat this acetone mixture to about 56 Celsius, and then gently stir the mixture for about 10 minutes. After 10 minutes, remove the heat source, and allow the mixture to cool to room temperature. Then filter-off the insoluble solids, and then vacuum dry or air-dry these filtered-off solids. Note: the acetone in both cases, can be recycled or discarded if desired. Then, place the dried filtered-off solids (in ether case) into a suitable beaker, and then add in 2 grams of anhydrous

sodium carbonate, followed by 2 grams of sodium chloride, and then manually blend the mixture for a few minutes to form a uniform mix. Then, slowly add in, in small portions at a time, a small amount of dry 99% isopropyl alcohol (the exact amount may vary), and rapidly stir the entire mixture during the addition of the alcohol until a fine paste is achieved. Note: only add in enough alcohol to form a uniform, thick paste—not a runny paste. After the addition of the alcohol, rapidly stir the entire alkaline alcoholic paste mixture for about 30 minutes. After 30 minutes, treat this alcoholic paste with 10 milliliters of toluene or xylene, and then thoroughly blend the entire mixture for about 10 minutes. After 10 minutes, allow the entire mixture to stand for about 5 minuets, and then filter-off the insoluble solids—note: collect the filtered solvent (the toluene or xylene) and set aside for just a moment. Thereafter, repeat this toluene or xylene treatment two more times using 10 milliliters of fresh toluene or xylene each time, and after each treatment, allow the mixture to stand for about 5 minuets, and then filter-off the insoluble solids. Note: after each filtration, collect the filtered solvent (the toluene or xylene), and after the third treatment, combine all three solvent portions (the toluene or xylene), if not already done so. Note: the filtered-off insoluble solids at this point can now be discarded if desired. Now, wash this combined toluene or xylene solvent portion with one 20-milliliter portion of warm water, followed by one 20-millilter portion of cold water, and then followed by another one 20-milliliter portion of warm water. After each washing portion, the toluene or xylene solvent portion will be the upper layer each time. After the washing process, dry the washed collected toluene or xylene portion by adding to it, 5 grams of anhydrous sodium sulfate, and then stir the entire solvent mixture for about 10 minutes—thereafter, filter-off the sodium sulfate. Finally, place this dried toluene or xylene portion into a shallow pan (with a high surface area), and allow the toluene or xylene solvent to evaporate. Note: blowing air over the surface of the toluene or xylene solvent mixture using a conventional cooling fan can be used to speed up the evaporation process. When all the solvent has been removed, collect the left over remaining crystals of pseudoephedrine, and then place these crystals into a suitable amber glass bottle, and store in a cool dry place until use.

Process 1B: Preparation of pseudoephedrine hydrochloride

Place the collected crystals of the freebase pseudoephedrine (obtained at the end of process 1A above), into a small beaker, and then add in 50 milliliters of dry diethyl ether. Thereafter, stir the entire mixture to dissolve all solids. Then place this ether mixture into an ice bath, and chill to about 0 Celsius. Thereafter, bubble into this mixture, 5 grams of dry hydrogen chloride gas (excess). After the addition of the hydrogen chloride, allow the entire ether mixture to stand at 0 Celsius for about 1 hour. After 1 hour, filter-off the precipitated crystals of pseudoephedrine hydrochloride, and then wash these crystals with one 20-millilter portion of fresh dry diethyl ether, and then vacuum dry or air-dry the crystals. Note: other salts of the freebase pseudoephedrine, such as the sulfate or phosphate, can be obtained by simply replacing the hydrogen chloride gas with the corresponding acid.

Intermediate-0008. Methedrine. *1-Phenyl-2-methyl-amino-ethan-1-ol*



Methedrine is the sister of ephedrine, and forms white to off-white crystals that may have a waxy feel or soapy feel to the touch. Methedrine can be used in the preparation of amphetamine derivatives utilizing similar reduction techniques as used for ephedrine.
Note: This substance is not a controlled substance, but pressure by anti-drug groups on legislation may have lead to new regulations. Check your local listings for potential regulations in regards to this substance. It will be concluded that this substance most likely will become more regulated due to its future role in the preparation of amphetamine derivatives. Note: Because of the non-existent use of this product in any commercial products, it is unlikely this compound has any form of regulation to it, as of yet. Note: the listing of this substance in this book is primarily for reference only. This compound when reduced in a similar manner as for ephedrine (see vide supra), produces a phenyl ethylamine derivative with no psychological activity. However, this compound can be used a "primer" for students and experimenters to produce halogenated derivatives of said compound. Methedrine can also be nitrated, chlorinated, and then reduced to the halogen containing amine by using standard nitro reduction techniques such as lithium aluminum hydride, iron/HCl, Tin/HCl, and the usual hydrogenation processes. It is recommend that this compound be experimented with, as this compound itself is not illegal, nor a "watch" substance. Experimentation with methedrine can help students and potential druggists practice their chemistry skills.

Procedure A: Preparation of methedrine (DL and L forms)

Materials:

1. 53 grams of benzaldehyde	7. 7 grams of a 37% formaldehyde solution
2. 30.5 grams of nitromethane	8. 6 grams of finely divided zinc
3. 30 milliliters of a 30% potassium carbonate solution	9. 6 grams of glacial acetic acid
4. 650 milliliters of diethyl ether	10. 6 grams of hydrogen sulfide gas
5. 90 milliliters of a 10% sodium bisulfite solution	11. 10 milliliters of 35 to 38% hydrochloric acid
6. 30 grams of anhydrous magnesium sulfate	12. 6 grams of anhydrous sodium carbonate

Summary: Methedrine is prepared in an identical manner as for ephedrine, with the nitroethane being replaced by nitromethane. For related information, see Serial number, 88,224, April 1st, 1916 by Wilhelm Nagajoshi, of Tokyo Japan, to M. Dick Bunnell, of San Francisco, CA; also see Serial number, 433,816, March 6th, 1930, by Chogi Nagai of Tokyo, Japan, to Alexander Nagai, of Berlin Germany.

Hazards: Use care when handling nitromethane, and diethyl ether, both of which form explosive mixtures with air and are highly flammable. Use caution when handling hydrogen sulfide gas, which is very toxic and flammable. Use maximum ventilation when handling concentrated formaldehyde solution, and avoid inhalation. Wear gloves when handling concentrated acetic acid, and hydrochloric acid, as they are both corrosive and can cause irritation.

Procedure:

Personnel notes for procedure A: Methedrine

Step 1: Preparation of phenylnitroethanol

Into a suitable beaker or flask, place 53 grams of benzaldehyde, followed by 30.5 grams of nitromethane. Immediately thereafter, add in 30 milliliters of a 30% potassium carbonate solution, and rapidly stir the entire mixture at room temperature for 2 hours. Note: A cold-water bath may or may not be needed to keep the reaction mixture at ambient temperature (room temperature). Do not allow the reaction mixture to get above 25 Celsius. After stirring for 2 hours, add to the reaction mixture 200 milliliters of diethyl ether, and shortly thereafter, add in 90 milliliters of a 10% sodium bisulfite solution, and then moderately stir the entire reaction mixture for 30 minutes. Afterwards, place the entire reaction mixture into a seperatory funnel, and then remove the upper ether layer (after removing the lower aqueous layer). Thereafter, wash this upper ether layer with three 75-milliliter portions of cold water. Note: after each washing portion, use a seperatory funnel to recover the ether layer, which will be the upper layer each time. After the washing portion, add to the ether layer, 15 grams of anhydrous magnesium sulfate (to absorb water), and then stir the entire mixture for 10 minutes. Then filter-off the magnesium sulfate. Then, place the filtered ether mixture into a distillation apparatus, or rotary evaporator, and remove the ether. When no more ether passes over or is collected, remove the remaining oily residue (after allowing it to cool to room temperature), and then place aside for step 2. This oily residue will consist of the desired phenylnitroethanol.

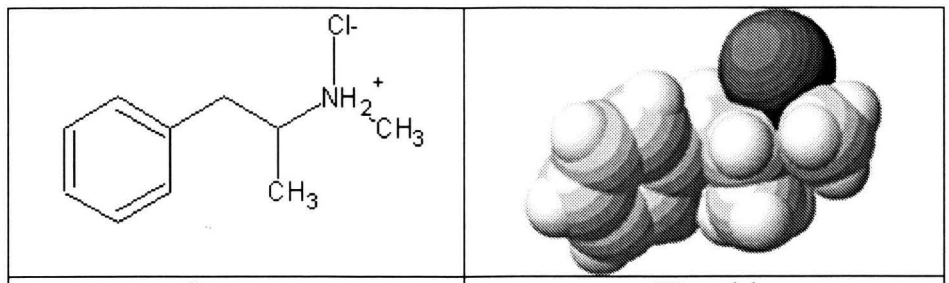
Step 2: Into a suitable flask equipped with motorized stirrer, and gas inlet tube, place 15 grams of the oily residue obtained in step 1, and then place this flask into an ice bath, and chill to 0 Celsius. When the oily residue from step 1 reaches a temperature of about 0 Celsius, add in 7 grams of a 37% formaldehyde solution. Thereafter, begin to stir these contents vigorously, and then carefully add in an acetic acid solution (prepared by adding and dissolving 6 grams of glacial acetic acid into 14 milliliters of ice cold water), and then carefully add in 6 grams of finely divide zinc (preferable zinc dust). During the addition of the acetic acid solution and zinc, vigorously stir the reaction mixture, and maintain its temperature below 5 celsius at all times. After the addition of the zinc, continue to stir the reaction mixture below 5 Celsius for about 30 minutes to complete the reaction. After 30 minutes, stop stirring, and then filter-off any insoluble zinc or other materials. Thereafter, place the filtered reaction mixture back into the ice bath, and then bubble into this filtered reaction mixture, 6 grams of hydrogen sulfide gas. Note: during the addition, vigorously stir the reaction mixture. Note: the addition of the hydrogen sulfide will precipitate any dissolved zinc. After the addition of the hydrogen sulfide, filter-off any insoluble materials, and then add to this filtered mixture, 250 milliliters of cold water, followed by 150 milliliters of diethyl ether, followed by 10 milliliters of a 35 to 38% hydrochloric acid solution. Then vigorously stir this entire mixture for about 1 hour. After 1 hour, place the entire reaction mixture into a seperatory funnel, and remove the lower water layer (which will contain the desired methedrine product as the hydrochloride). Note: the upper ether layer can be discarded or recycled if desired. Now, to the recovered lower water layer, add in a sodium carbonate solution prepared by adding and dissolving 6 grams of anhydrous sodium carbonate into 15 milliliters of cold water, and then stir the whole mixture for about 15 minutes. Finally, extract this entire mixture with three 100-milliliter portions of diethyl

ether, and after the extraction process (after each extraction, the ether will be the upper layer), combine all ether portions, if not already done so, and then dry this combined ether portion by adding in 15 grams of anhydrous magnesium sulfate. Then stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Thereafter, place this filtered ether mixture into a distillation apparatus or rotary evaporator, and remove the ether. When no more ether is recovered, recover the left over remaining residue (after it has cooled to room temperature), and then store in an appropriate amber glass bottle until use.

Note: this freebase methedrine will actually be a mixture of the DL and L-forms, from which the L-form is the most common used in the preparation of methamphetamine. However, regardless of other literature, amphetamines can be prepared using either the DL-form or the L-form with satisfactory results.

Note: Other salts of the freebase methedrine such as the sulfate, tartrate, citrate, and phosphate can be prepared by adding the corresponding acid to the freebase compound obtained at the end of step 2 (the freebase should be dissolved into ether in the ratio of 1 grams of freebase to 15 grams of diethyl ether). For the hydrochloride, 1 mole of acid gas should be bubbled into the ether mixture for every 1 mole of freebase methedrine (not for the total weight of the ether mixture). For sulfuric acid or tartaric acid, 1 mole of sulfuric acid or d-tartaric acid should be added for 2 moles of the freebase methedrine (not for the total weight of the ether mixture); and for citric acid or phosphoric acid, 1 mole of the citric acid or phosphoric acid should be added to 3 moles of the freebase methedrine (not for the total weight of the ether mixture). The ether mixture in each of these cases can be evaporated using a distillation apparatus, or rotary evaporator only to the point where 80% of the total volume is reduced. The resulting ether concentrate can then be filtered to recover the product, which can then be vacuum dried or air-dried. All the salts of methedrine are mild stimulants, but can be used in the preparation of stimulant compositions (by addition to amphetamines, or other CNS stimulants), or can be used in the preparation of other stimulants.

0009. Methamphetamine hydrochloride. *N-methyl-N-(1-methyl-2-phenylethyl)amine hydrochloride; speed; ice; crank;*



Structure	3D model

Methamphetamine hydrochloride forms colorless to white crystals, which may be colored orange to brown depending on purity. The pure crystals have a melting point of about 170 Celsius, but the crystals may melt at 175 Celsius depending on purity. The crystals have a bitter taste, with a strange after taste. Methamphetamine hydrochloride is soluble in water, alcohol, and chloroform, but insoluble in ether. The crystals when dissolved in water, show a moderate acid reaction to litmus. Note: for best effects, methamphetamine hydrochloride should not be used in combination with alcohol, and users of said product should consume large volumes of water while under the influence to prevent any series medical problems such as dehydration.
Note: This substance is a controlled substance (stimulant) as listed in the US code of Federal regulations.

Toxicity: Moderate	**Rate of onset (average):** Rapid
Stimulation dosage (ingestion): 40 to 50 milligrams	**Duration of stimulation (average):** 8 to 12 hours (depending on the person)
Stimulation dosage (inhalation): 30 to 35 milligrams	**Habit forming potential:** High
Stimulation dosage (injection): 12 milligrams +	**Estimated value U.S. (based on procedure):** $20 per gram

Procedure A: Preparation of methamphetamine hydrochloride

Materials:

1. 50 grams of 46% hydroiodic acid (hydriodic acid)	5. 450 milliliters of diethyl ether
2. 15 grams of ephedrine (freebase) or 15 grams of pseudoephedrine (freebase)	6. 15 grams of anhydrous magnesium sulfate
3. 4 grams of red phosphorus	7. 3.6 grams of dry hydrogen chloride gas
4. 50 grams of powdered anhydrous sodium carbonate	

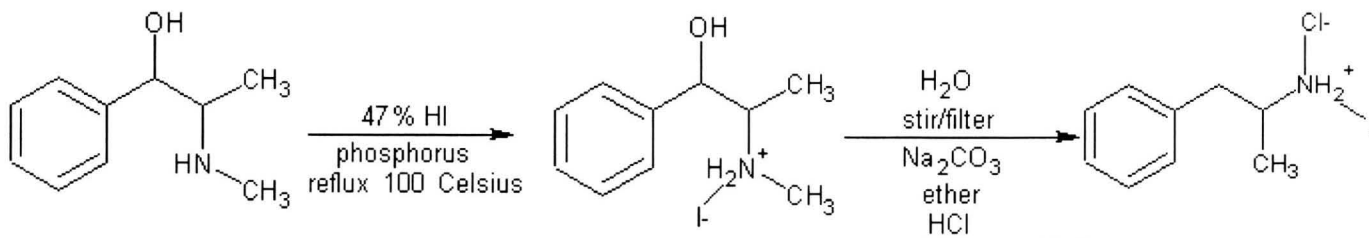
Summary: Methamphetamine hydrochloride is readily prepared by reducing ephedrine or pseudoephedrine with hydroiodic acid (commonly called hydriodic acid) and red phosphorus under reflux. The reaction is generally simple, and afterwards, the reaction mixture is allowed to cool, whereby water is added to dilute the mixture, and the resulting dilute aqueous mixture is then filtered, and treated with excess sodium carbonate to neutralize any acid salts, and acid products. The resulting alkaline solution is then extracted with ether, and the collected ether extracts are then treated with hydrogen chloride gas to precipitate the desired product as the hydrochloride salt.

Hazards: Use caution when handling hydriodic acid, which is a strong oxidizing agent, and can cause skin burns. Note: store hydroiodic acid in amber glass bottles in a cool place. Hydriodic acid can deteriorate on standing. Use great care when handling diethyl ether, which can form explosive mixtures with air—extinguish all flames before using.

Procedure:

Personnel notes for procedure A: Methamphetamine hydrochloride

Into a suitable reflux apparatus (equipped with motorized stirrer, and thermometer), place 15 grams of freebase ephedrine (either form will work including pseudoephedrine), and then slowly add in 50 grams of 46% hydriodic acid (hydroiodic acid), and then stir the entire mixture for about 10 minutes. Note: during the addition, do not allow the temperature to rise above 40 Celsius. After stirring for 10 minutes, rapidly add in 4 grams of finely divided red phosphorus, and then reflux the entire reaction mixture at about 100 Celsius for 24 hours. During the reflux period, rapidly stir the reaction mixture. After refluxing the reaction mixture for 24 hours, remove the heat source, and then allow the reaction mixture to cool to room temperature. Thereafter, pour the entire reaction mixture into a clean suitable beaker, and then add in 250 milliliters of cold water, and then stir the entire mixture for 10 minutes. After 10 minutes, filter the entire reaction mixture, and then place the filtered reaction mixture into a clean beaker, and then gradually add in 50 grams of powdered anhydrous sodium carbonate, and then stir the entire reaction mixture for about 1 hour at room temperature. Then, extract the entire reaction mixture with six 75-milliliter portions of diethyl ether, and after the extraction process, combine all ether extracts (if not already done so), and then dry the combined ether portion by adding in 15 grams of anhydrous magnesium sulfate—then stir the entire mixture for 10 minutes, and then filter-off the magnesium sulfate. Then place this filtered ether mixture into an ice bath, and chill to about 5 Celsius. Thereafter, bubble into the ether mixture, 3.6 grams of dry hydrogen chloride gas, and then after the addition of the acid gas, allow the entire ether mixture to stand for 30 minutes at 5 Celsius. Thereafter, filter-off the precipitated product, and then vacuum dry or air-dry this precipitated product. Note: the ether mixture can be evaporated to yield a small amount of additional product. Note: For future other synthesis utilizing the freebase methamphetamine, the freebase methamphetamine compound can be collected by simply distilling-off the ether (of the combined ether extracts) instead of adding the hydrogen chloride gas.



Ephedrine

Methamphetamine hydrochlor

Note: Other salts of the freebase methamphetamine such as the sulfate, tartrate, citrate, and phosphate can be prepared by adding the corresponding acid to the ether mixture of the methamphetamine freebase compound obtained at the end of the above process. For sulfuric acid or tartaric acid, 4.2 grams of 98% sulfuric acid or 6.5 grams d-tartaric acid should be added to the ether mixture of the methamphetamine freebase. For citric acid or phosphoric acid, 5.5 grams of citric acid or 2.8 grams of phosphoric acid should be added to the ether mixture of the methamphetamine freebase. The ether mixture after treatment with the corresponding acid in each of these cases can then be evaporated using a distillation apparatus, or rotary evaporator to the point where only 80% of the total volume of the ether mixture has been reduced. The resulting ether concentrate can then be filtered to recover the product, which can then be vacuum dried air-dried. All the salts of methamphetamine are powerful stimulants.

Procedure B: Preparation of methamphetamine hydrochloride (direct process; iodine process)

Materials:

1. 75 milliliters of glacial acetic acid	6. 200 grams of anhydrous sodium carbonate
2. 7.5 grams of red phosphorus	7. 300 milliliters of diethyl ether
3. 16.7 grams of iodine crystals	8. 20 grams of anhydrous magnesium sulfate
4. 4 grams of ephedrine hydrochloride (see note in procedure) or 4 grams of pseudoephedrine hydrochloride (see intermediate-007-03)	9. 2 grams of dry hydrogen chloride gas
5. 10 grams of sodium bisulfite	

Summary: Methamphetamine hydrochloride can be prepared in a modified process whereby ephedrine hydrochloride or pseudoephedrine hydrochloride is reacted with a reaction mixture containing the intermediate product from the reaction of red phosphorus and iodine with glacial acetic acid. The reaction is rather moderate, and afterwards, the mixture is heated, diluted with water, made alkaline, and then extracted with ether in the usual manner. The concentrated ether mixture is then treated with hydrogen chloride to precipitate the desired product as the hydrochloride.

Hazards: Extinguish all flames before using diethyl ether, which can form explosive mixtures with air. Handle iodine with caution, as it is capable of producing irritating and corrosive vapors. Glacial acetic acid is corrosive and capable of producing skin irritation, so wear gloves when handling. Use proper ventilation when handling hydrogen chloride gas, which is very irritating and corrosive.

Procedure:

Personnel notes for procedure B: Methamphetamine hydrochloride

Into a suitable flask, add 75 milliliters of glacial acetic acid, followed by 7.5 grams of red phosphorus, and then followed by 16.7 grams of iodine crystals. Thereafter, stir the entire mixture for about 15 minutes, and monitor the temperature to make sure it does not rise above 40 Celsius. After 15 minutes, much of the iodine will have reacted with the glacial acetic acid, if however, it appears a chemical reaction is still taking place after the initial 15 minutes, continue to stir the mixture for an additional 15 minutes. Thereafter, add in 25 milliliters of cold water, and then stir the entire mixture for about 10 minutes. Then, add in 4 grams of ephedrine hydrochloride or 4 grams of pseudoephedrine hydrochloride, and then stir the entire reaction mixture for about 30 minutes. Thereafter, pour the entire reaction mixture into a reflux apparatus, or simply attach a reflux condenser to the flask being used for the reaction, and then gently reflux (merely gently heat), the entire reaction mixture at 80 to 90 Celsius for about 90 minutes. Note: during the reflux operation (heating period), gently stir the reaction mixture. **Note: the ephedrine hydrochloride employed in this reaction can be obtained by dissolving the freebase ephedrine into ether (ratio of 1 gram of freebase ephedrine to 15 grams of diethyl ether, chilling the resulting ether mixture to 0 Celsius, and then bubbling dry hydrogen chloride gas there into (1 mole of hydrogen chloride per 1 mole of freebase ephedrine), followed by distillation to remove the ether only until 80% of the total volume has been reduced. Then, when most of ether has been removed, filter-off the precipitated ephedrine hydrochloride (after the ether concentrate has cooled to room temperature), and then vacuum dry or air-dry it.** After the reflux period, remove the heat source, and allow the reaction mixture to cool to room temperature. Then add to it, 150 milliliters of cold water, and then stir the entire mixture for about 10 minutes, and then filter-off any insoluble materials. Now, gradually add to the filtered reaction mixture, a sodium bisulfite solution prepared by adding and dissolving about 10 grams of sodium bisulfite into 500 milliliters of water. During the addition of this sodium bisulfite solution, rapidly stir the reaction mixture. After the addition of the sodium bisulfite solution (to destroy impurities), add in a sodium carbonate solution prepared by adding and dissolving 200 grams of anhydrous sodium carbonate into 750 milliliters of water. After the addition of the sodium carbonate solution, rapidly stir the entire reaction mixture for 1 hour at ambient temperature (room temperature). After 1 hour, extract the entire reaction mixture with four 75-milliliter portions of diethyl ether. After the extraction process, combine all ether extracts (if not already done so), and then dry this combined ether portion by adding to it 20 grams of anhydrous magnesium sulfate, and then stir the entire ether mixture for about 10 minutes. Thereafter, filter-off the magnesium sulfate, and then place this filtered ether mixture into an ice bath, and chill to about 0 Celsius. Thereafter, bubble into the chilled ether mixture, 2 grams of dry hydrogen chloride gas. After the addition of the hydrogen chloride, filter-off the precipitated methamphetamine hydrochloride product, and then vacuum dry or air-dry the product. Note: the filtered ether mixture can be evaporated to recover a small amount of additional product.

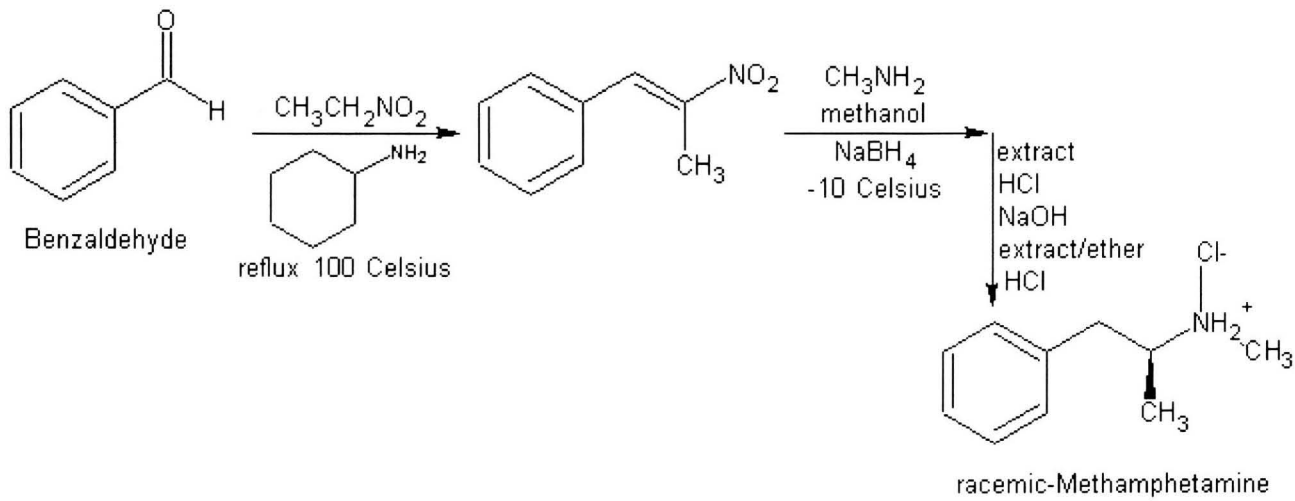
Note: Other salts of the freebase methamphetamine such as the sulfate, tartrate, citrate, and phosphate can be prepared by adding the corresponding acid to the ether mixture of the methamphetamine freebase compound obtained at the end of the above process. For sulfuric acid or tartaric acid, 1 gram of 98% sulfuric acid or 1.5 grams d-tartaric acid should be added to the ether mixture of the methamphetamine freebase. For citric acid or phosphoric acid, 1.2 grams of citric acid or 600 milligrams of phosphoric acid should be added to the ether mixture of the methamphetamine freebase. The ether mixture after treatment with the corresponding acid in each of these cases can then be evaporated using a distillation apparatus, or rotary evaporator to the point where only 80% of the total volume of the ether mixture has been reduced. The resulting ether concentrate can then be filtered to recover the product, which can then be vacuum dried or air-dried. All the salts of methamphetamine are powerful stimulants.

Procedure C: Preparation of racemic-methamphetamine hydrochloride (ICE)

Materials:

1. 27 grams of benzaldehyde	8. 30 grams of anhydrous sodium sulfate
2. 20 grams of nitroethane	9. 3.75 grams of sodium borohydride
3. 5 milliliters of cyclohexylamine	10. 200 milliliters of methylene chloride
4. 300 milliliters of dry hexane	11. 60 grams of dry hydrogen chloride gas
5. 500 milliliters of dry methanol	12. 50 grams of sodium hydroxide
6. 25 grams of methylamine gas	13. 450 milliliters of diethyl ether
7. 55 grams of anhydrous magnesium sulfate	

Summary: racemic-Methamphetmaine (ice), is prepared by reacting phenyl-2-nitropropene with methylamine and sodium borohydride in the presence of methanol. The phenyl-2-nitropropene is formed by the condensation of benzaldehyde with nitroethane in the presence of cyclohexylamine. After the reaction of phenyl-2-nitropropene with sodium borohydride and methanol, the reaction mixture is treated with methylene chloride, and this resulting methylene chloride mixture is then treated with hydrogen chloride, and the resulting acidified mixture is then evaporated to remove the bulk of the methylene chloride. After the bulk of the methylene chloride has been removed, the left over remaining concentrate is then filtered to recover the precipitated product. The precipitated product is then treated with sodium hydroxide to liberate the freebase, which is then extracted into ether. The ether extract is then treated with hydrogen chloride to precipitate the ICE, which is then filtered-off and then vacuum dried or air-dried.



Hazards: Extinguish all flames before using diethyl ether and hexane, which are highly flammable, and capable of forming explosive mixtures with air. Methanol burns with a colorless flame, so extinguish all flames before handling. Use care when handling sodium borohydride, which reacts violently with water generating excessive heat.

Procedure:

Personnel notes for procedure C: racemic-Methamphetamine hydrochloride

Step 1: Preparation of phenyl-2-nitropropene

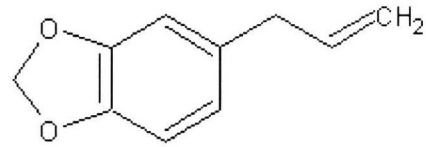
Into a suitable reflux apparatus, place all at once, 27 grams of benzaldehyde, followed by 20 grams of nitroethane, followed by 5 milliliters of cyclohexylamine. Thereafter, reflux the entire mixture at about 100 Celsius for 3 hours. After refluxing for 3 hours, remove the heat source, and allow the two-phase reaction mixture to cool to room temperature. Then pour the entire reaction mixture into a seperatory funnel, and remove the lower (organic) layer. The upper layer can be recycled or discarded if desired as it will contain the cyclohexylamine catalyst. Then place the recovered lower organic layer into a suitable sized beaker, and then add in 25 milliliters of cold water. Immediately thereafter, rapidly stir the mixture using magnetic stirrer, or other means, for about 30 minutes at room temperature. Then remove the upper water layer by decanting it off, and then place the lower organic layer (containing the desired product), into an ice bath and chill to about 0 Celsius. Then, add in about 10 milliliters of cold water, and then allow the total mixture to stand at room temperature for several hours to allow the desired product of phenyl-2-nitropropene to crystallize. After 2 hours, most of the desired nitro compound should have precipitated and afterwards, filter-off the precipitated crystals, and then vacuum dry or air-dry them. Finally, recrystallize these dried collected crystals from 150 milliliters of dry hexane, and after the recrystallization process, vacuum dry or air-dry the crystals.

Step 2: Preparation of racemic-methamphetamine

Into a suitable beaker, place 250 milliliters of dry methanol, and then bubble into this methanol, 25 grams of methylamine gas. After the addition of the methylamine gas, add to the resulting methylamine/methanol solution, 25 grams of anhydrous magnesium sulfate (to absorb water), and then place this entire mixture into a suitable flask, and then stopper the flask. Immediately thereafter, stir the entire mixture for about 10 minutes. After 10 minutes, carefully filter-off the magnesium sulfate, and do it as fast as possible to avoid moisture absorption by the methylamine/methanol mixture. After the filtration, place the methylamine/methanol mixture into a suitable 3-neck flask (equipped with motorized stirrer, thermometer, and addition funnel), and then place a phenyl-2-nitropropene solution into the addition funnel—this solution being prepared by adding and dissolving 34 grams of phenyl-2-nitropropene (obtained in step 1) into 150 milliliters of dry hexane. Note: the 3-neck reaction flask should be equipped with a calcium chloride drying tube to keep moisture from entering the apparatus. Then place the 3-neck reaction flask into a cold-water bath at about 10 to 15 Celsius. Then add to the methylamine/methanol mixture, 30 grams of anhydrous sodium sulfate. Note: this sodium sulfate is to absorb any water formed during the reaction. Note: dried silica gel pieces can be used instead of sodium sulfate if desired. Now, slowly add drop-wise, the phenyl-2-nitropropene/hexane solution form the addition funnel, to the methylamine/methanol mixture over a period sufficient to keep the reaction mixture below 25 Celsius at all times. During the addition, moderately stir the reaction mixture with the motorized stirrer. After the addition of the nitropropene/hexane solution, continue to stir the reaction mixture for 1 hour at a temperature below 25 Celsius. After this additional 1 hour of mixing, stop stirring, and then quickly filter the reaction mixture to remove the insoluble sodium sulfate. Then place this filtered reaction into a clean flask, and then place this flask into an ice bath, and chill to –10 Celsius. Note: while waiting for the reaction mixture to chill, stopper the flask to keep moisture out. When the temperature of the reaction mixture reaches –10 Celsius, remove the stopper, and replace it with a standard powder funnel, and then slowly add in, in small portions at a time, 3.75 grams of sodium borohydride, and after each portion, add in 25 milliliters of methanol (ten 375-milligram portions of sodium borohydride and ten 25-milliliter portions of methanol). During the entire addition, rapidly stir the reaction mixture, and maintain its temperature below 20 Celsius at all times. After the addition of the sodium borohydride and methanol portions, continue to stir the entire reaction mixture at a temperature below 20 Celsius for about 3 hours. After 3 hours, pour the entire reaction mixture into a large flask, and then add in 1200 milliliters of water. Shortly thereafter, add in 200 milliliters of methylene chloride, and then rapidly stir the reaction mixture for about 30 minutes at room temperature. Then decant-off (pour-off) the upper aqueous layer, and then place the remaining lower methylene chloride layer into a seperatory funnel, and drain-off the lower methylene chloride layer—as there will be some upper aqueous layer still remaining. Thereafter, place the methylene chloride layer into a beaker, and then add in 15 grams of anhydrous magnesium sulfate (to absorb water). Then stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Then, place the entire mixture into a suitable sized beaker, and then bubble into the mixture, 30 grams (excess) of dry hydrogen chloride gas. After the addition of the hydrogen chloride gas, place the entire acidified mixture into a distillation apparatus or rotary evaporator, and distill-off the methylene chloride at 40 Celsius until only 80% of the total volume remains. Once this point is reached, stop the distillation process, and then remove the left over remaining contents (after it has cooled to room temperature), and then filter these contents to recover the precipitated impure product. Then vacuum dry or air-dry the filtered-off crystals. Thereafter, place these crystals into a clean beaker, and then add in a sodium hydroxide solution prepared by adding and dissolving 50 grams of sodium hydroxide into 250 milliliters of water, and after the addition, stir the entire mixture for about 30 minutes at room temperature. Note: sodium hydroxide generates much heart when dissolved in water, so allow the solution to cool before using. After stirring the alkaline mixture for about 30 minutes, extract the entire mixture with three 150-millilter portions of diethyl ether, and after the extraction, combine all ether portions (if not already done so), and then dry this combined ether portion by adding to it, 15 grams of anhydrous magnesium sulfate. Then stir the entire mixture for

about 10 minutes, and then filter-off the magnesium sulfate. Finally, place the filtered ether mixture into an ice bath, and chill to 0 Celsius. Thereafter, bubble into the ether mixture, 30 grams of dry hydrogen chloride gas (excess), and after the addition, stir the entire ether mixture for about 30 minutes. Thereafter, filter-off the precipitated crystals, and then vacuum dry or air-dry the crystals.

Intermediate-0010. Safrole. *5-allyl-1,3-benzodioxole*



Safrole forms a colorless to slightly yellow liquid with the odor of sassafras. The oil is insoluble in water, but very soluble in alcohol, and miscible with chloroform and ether. The oil has a boiling point of 232 Celsius, but can be distilled under high vacuum at 100 Celsius under 11 millimeters of mercury. Safrole is the main component of sassafras oil, from which is makes up 70 to 75% of the oil by weight. Safrole also exists in Ocotea cymbarum oil (Brazilian oil of sassafras), which it exists up to 90% by weight. The oil of massoria bark and Cinnamomum massoia contains about 14% safrole. Safrole can be extracted from sassafras oil by the means described later, and it can be extracted from Massoria bark oil and Cinnamomum massoia by washing the corresponding oil with sodium hydroxide solution to remove the phenols, and then vacuum distilling to obtain the safrole boiling at about 100 celsius under a vacuum of 11 millimeters of mercury, or by carefully fractionally distilling (two path distillation) the phenol free oil at 228 to 235 Celsius. Safrole can also be made synthetically from rather inexpensive reagents (see procedure B below). Sassafras oil can be obtained by steam distilling the ground up roots of the sassafras tree, which grows in the mid western United States. Other sassafras species of tress elsewhere in the world can also be used to obtain the safrole by steam distillation from the root. To identify a sassafras tree, consult a book that discusses the various types of trees and plants. The dried root bark of the sassafras tree contains about 10% safrole by weight, and the remainder of the root contains only about 1%. The dried root bark can be obtained from numerous sources, including herb stores, health food suppliers, and botanical suppliers. Sassafras oil can also be obtained from these aforementioned sources; if however your local suppliers do not offer the sale of sassafras oil, request them to order some for you, which they should have no problem doing. Safrole is also used in perfumes, so check out the types of perfumes, and their ingredients. Note: checkout your local aromatherapy suppliers, as they are major consumers of oils, one of which may be sassafras oil. Sassafras oil may be used in adulterants in massage oils for use in aromatherapy. Ocotea cymbarum oil is obtained by steam distillation of the wood of the Ocotea pretiosa tree, which grows in South America. The wood contains about 1% oil by weight, which is easily collected by steam distillation of the wood chips, and the resulting steam distilled product contains about 90% safrole by weight. Distributors of perfume and flavoring compounds may contain this Ocotea cymbarum oil. Check the OPD directory for essential oils and botanical companies; also checkout small herb shops nationwide.

Procedure A: Extraction of safrole from sassafras oil

Procedure:

Personnel notes for procedure A: Safrole

Note: as previously mentioned, sassafras oil can be obtained from the root bark of the sassafras tree. To do this, setup a standard steam distillation apparatus, and then steam distill the root bark (grind the root bark into pieces before use). The oil and water collect in the receiver flask, where upon the oil can be seen as droplets. The oil is denser then water so it will form droplets below the water. After the steam distillation process, the oil can be collected by placing the water/oil mixture into a seperatory funnel, and then recovering the lower oil layer. The collected oil layer should then be dried by mixing with it, a small amount of anhydrous calcium chloride. After filtering-off the calcium chloride, place the oil into a vacuum distillation apparatus, and vacuum distill at 100 Celsius under a vacuum of 11 millimeters of mercury (see figure below). Note: other oils containing safrole can be vacuum distilled in a similar manner. Note: because of the expense involved in purchasing vacuum distillation apparatus, try freezing the sassafras oil, or other oils that contain the safrole. Safrole has a melting point of 11

Celsius, and it may be possible to crystallize the safrole out of any oil solution by using ice baths, cold-water baths, or even a freezer. Experiment with various techniques; solvent extractions may also work.

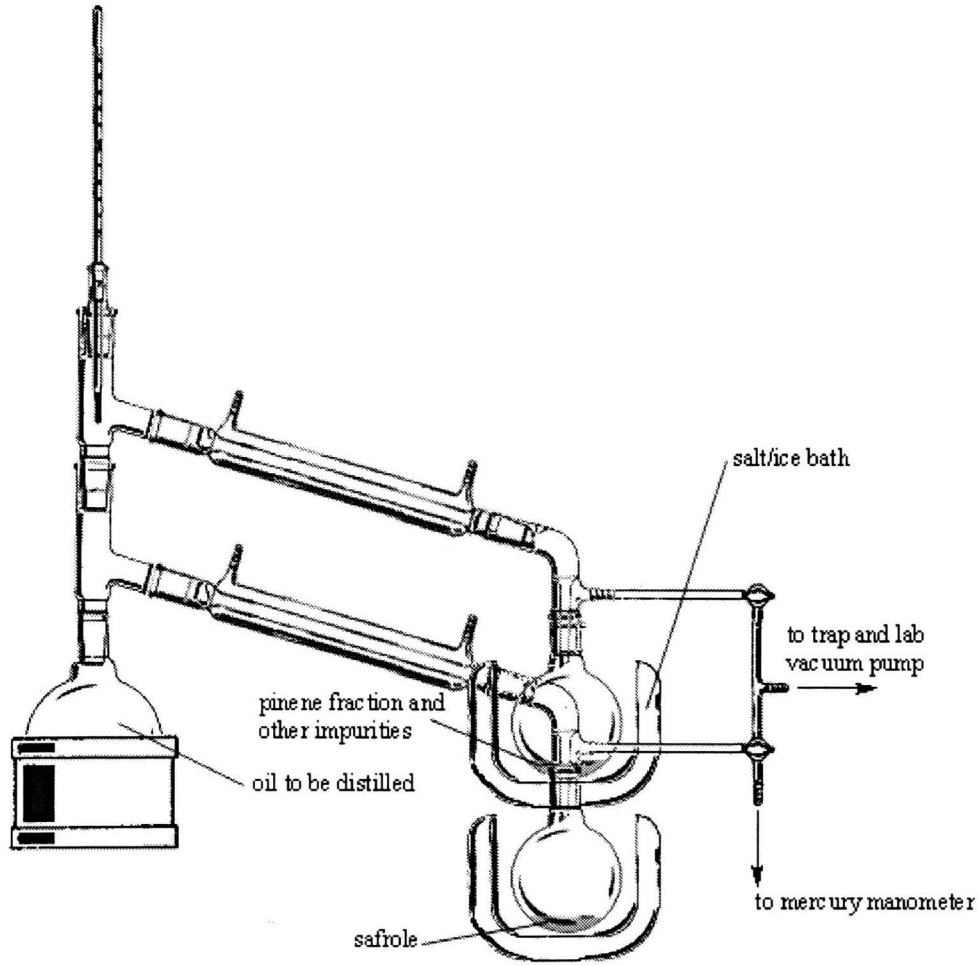


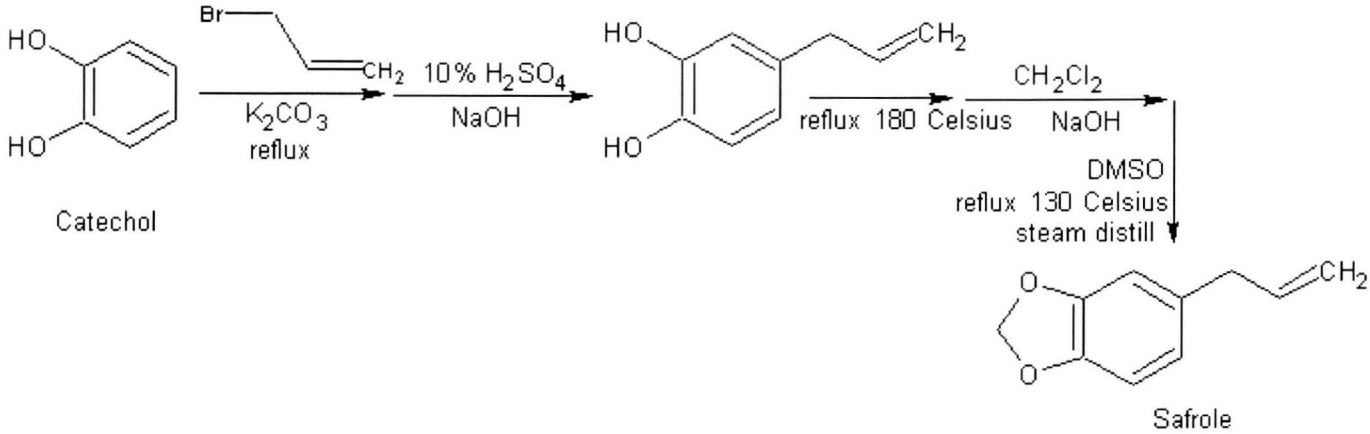
Figure 042. Two-path vacuum distillation apparatus for collecting safrole Note: in some cases, the safrole may be the upper fraction, depending on density, and impurities.

Procedure B: Synthesis of safrole from catechol

Materials:

1. 13 grams of catechol	6. 50 milliliters of diethyl ether
2. 14 grams of allyl bromide	7. 76.5 grams of sodium hydroxide
3. 22 milliliters of dry acetone	8. 200 milliliters of methylene chloride
4. 17 grams of finely divided anhydrous potassium carbonate	9. 15 grams of anhydrous magnesium sulfate
5. 200 milliliters of 10% sulfuric acid solution	

Summary: Safrole can be synthetically prepared by methylating 4-allyl catechol. This 4-allyl catechol is prepared by reacting catechol with allyl bromide, followed by an exhaustive extraction process, and then followed by a pain staking distillation process. Once the 4-allyl catechol has been successfully obtained, it is converted into safrole by refluxing it with methylene chloride and dimethyl sulfoxide in the presence of sodium hydroxide. The resulting reaction mixture is then distilled to remove any unreacted methylene chloride, and the resulting mixture is then steam distilled to collect the oily safrole product, which is then collected via a seperatory funnel.



Hazards: Extinguish all flames before using diethyl ether, which is highly flammable, and can form explosive mixtures with air. Use care when handling acetone, which is also highly flammable. Use maximum ventilation when handling allyl bromide, which is very irritating to the eyes, nose, and throat.

Procedure:

Personnel notes for procedure B: Safrole

Step 1: Preparation of 4-allyl catechol

Into a suitable reflux apparatus, place 13 grams of catechol, followed by 14 grams of allyl bromide, and then add in 22 milliliters of dry acetone. Then stir the entire mixture to form a uniform mixture. Immediately thereafter, gradually add in 17 grams of finely divided anhydrous potassium carbonate, and stir the mixture while adding this potassium carbonate. After the addition of the potassium carbonate, reflux the entire reaction mixture at 60 Celsius for about 3 hours. Note: fit a calcium chloride drying tube to the top of the reflux condenser to keep moisture out from the apparatus. After refluxing for about 3 hours, quickly remove the reflux condenser, and replace it with a conventional cold water condenser, fitted with a receiver flask, and then distill-off the acetone until no more acetone passes over into the receiver flask. When this point is reached, stop the distillation process, and allow the reaction mixture to cool to room temperature. Thereafter, pour the distilled reaction mixture left over, into a clean beaker, and then add in 25 milliliters of cold water, followed by 100 milliliters of 10% sulfuric acid solution. Then stir the entire acidic reaction mixture for about 10 minutes. Thereafter, extract the entire reaction mixture with one 50-milliliter portion of diethyl ether. After the extraction process, wash the ether portion by adding to it, a sodium hydroxide solution prepared by adding and dissolving 35 grams of sodium hydroxide into 150 milliliters of water. Note: the addition of sodium hydroxide to water generates much heat, so allow the mixture to cool to room temperature before using. Thereafter, remove the upper ether layer by using a seperatory funnel, or by decantation, and then discard or recycle this upper ether layer (will contain diallyl ether). Now to the lower water layer, add in 100 milliliters of 10% sulfuric acid, and upon the acid addition, some oil should separate. After the addition of the sulfuric acid, extract the entire acidic mixture (including any separated oil) with three 50-milliliter portions of methylene chloride. Note: after each extraction, the methylene chloride will be the upper layer. After the extraction process, combine all methylene chloride extracts, if not already done so, and then dry this combined methylene chloride mixture by adding to it, 15 grams of anhydrous magnesium sulfate—thereafter, stir the whole mixture for about 10 minutes, and then filter-off the magnesium sulfate. Thereafter, place the filtered methylene chloride mixture into a distillation apparatus or rotary evaporator, and remove the methylene chloride. When no more methylene chloride is collected, recover the left over remaining oil. Now, to this oil, place it into a reflux apparatus, and heat it to 180 Celsius. Note: during the heating process, the oil will self heat raising the temperature to about 260 Celsius. When this temperature change results, stop the heating process, and then place the oil (which will now be red in color) into a vacuum distillation apparatus (after it has cooled, or simply replace the reflux condenser with the appropriate glass adapters and immediately begin the vacuum distillation process), as similar to the one used for the distillation of safrole as listed above, but use only one condenser and receiver rather then two, and distill the oil at 158 celsius under a vacuum of 16 millimeters of mercury. When no more oil is obtained at this temperature and vacuum, stop the distillation process, and then remove the left over remaining residue, and discard it. To the collected fraction, re-vacuum distill it using the same apparatus (after it has been

cooled, and cleaned), and re-vacuum distill the oil at 158 Celsius, under a vacuum of 16 millimeters of mercury to obtain a refined 4-allyl catechol product.

Step 2: Preparation of safrole by methylating the 4-allyl catechol

Into a suitable 3-neck flask fitted with motorized stirrer, reflux condenser, thermometer, and a mercury bubbler fitted into the left or right female joint of the 3-neck flask to form an apparatus that is excluded from the atmosphere. Note: the top of the reflux condenser should be fitted with a calcium chloride drying tube, and to the other end of the drying tube, a gas inlet tube connected to a cylinder of dry nitrogen should be in place. Before assembling the calcium chloride dying tube with nitrogen purge adapter, charge the 3-neck flaks with 50 milliliters of methylene chloride, followed by 250 milliliters of dimethyl sulfoxide (DMSO). Thereafter, begin the nitrogen purge and allow the entire apparatus to be flushed with nitrogen to exclude air and moisture—the mercury bubbler is to allow the nitrogen gas to run through the apparatus, and then escape into the atmosphere without exposing the apparatus to the atmosphere. Once the apparatus has been purged with nitrogen, reflux the contents in the 3-neck flask using a heating mantle to 130 Celsius, and when the temperature of these contents reaches the desired 130 Celsius mark, add in 3.7 grams of 4-allyl catechol (obtained in step 1), followed immediately by 2 grams of sodium hydroxide. Note: the 4-allyl catechol and sodium hydroxide can be quickly added through the top of the reflux condenser by quickly and temporarily removing the calcium chloride drying tube briefly, and then reattaching it after the addition. This process of addition should then be repeated 19 times (3.7 grams of 4-allyl catechol and 2 grams of sodium hydroxide each time for a total of 74 grams of 4-allyl catechol and 40 grams of sodium hydroxide). During each addition maintain the reflux at 130 Celsius. After the last addition, reflux the entire reaction mixture for an additional 10 minutes, and thereafter, add in 10 milliliters of methylene chloride followed by 1.5 grams of sodium hydroxide. Thereafter, continue to reflux the reaction mixture at 130 Celsius for 35 minutes. Finally, after refluxing for the final 35 minute time period, stop the heating and reflux, and then allow the entire reaction mixture to cool to room temperature. Then pour the entire reaction mixture into a distillation apparatus, and distil-off any unreacted methylene chloride. When no more methylene chloride distills over, allow the left over remaining contents to cool to room temperature before removing from the apparatus, and then place these contents into a steam distillation apparatus, and then add in 500 milliliters of water, and then steam distill the safrole from the mixture to obtain safrole and water in the receiver flask. The oily safrole layer can then be recovered by using a seperatory funnel in the usual manner. The safrole can be vacuum distilled at 100 Celsius under a vacuum of 11 millimeters of mercury to obtain a refined product if desired.

Procedure C: Synthesis of safrole from eugenol

Materials:

1. 115 milliliters of dry hexane	11. or 3 grams of eugenol (obtained in Intemreidate-0025. Eugenol)
2. 2.7 grams of aluminum foil	12. or 15 grams of anhydrous sodium sulfate
3. 38 grams of dry iodine	13. or 50 milliliters of toluene
4. 16 grams of eugenol (obtained in Intemreidate-0025. Eugenol)	14. 6 grams of potassium fluoride
5. 900 milligrams of n-butyl ammonium iodide	15. 100 milliliters of dimethylformamide (DMF)
6. 75 milliliters of dry ethyl acetate	16. 10 grams of methylene chloride
7. 75 milliliters of toluene	17. 150 milliliters of diethyl ether
8. or 10 grams of pyridine	18. 150 milliliters of a 5% sodium hydroxide solution
9. or 200 milliliters of diethyl ether	19. 15 grams of anhydrous sodium sulfate
10. or 5 grams of dry hydrogen chloride gas	

Summary: Safrole can be made from eugenol in a two step process, starting with the formation of 4-allylbenzene-1,2-diol. This 4-allylbenzene-1,2-diol intermediate is prepared by reaction of eugenol with aluminum iodide under reflux in the presence of a catalyst. The reaction is rather general, and afterwards, the desired 4-allylbenzene-1,2-diol can be obtained by filtration, followed by dissolving the filtered-off precipitate into ethyl acetate, filtering the ethyl acetate mixture to remove impurities, and then evaporating off the ethyl acetate to recover the crude product. The crude product can be recrystallized from toluene for purity. In a modified process, the intermediate 4-allylbenzene-1,2-diol can be obtained by reacting eugenol with pyridine hydrochloride under microwave heating. The reaction is generally simple, and after the microwave heating periods, the reaction mixture is quenched with water, and then extracted with ether. The combined ether extract is then evaporated to recover the crude residue of 4-allylbenzene-1,2-diol, which is then purified in the usual manner. The 4-allylbenzene-1,2-diol is then converted into the desired safrole by reaction with potassium fluoride and methylene chloride under high temperature. After the reaction period, the reaction mixture is quenched with cold water, and then extracted with ether. The combined ether portion is then washed, and then evaporated to yield the crude safrole product as a oily residue. This oily residue can be purified by vacuum distillation, solvent purification, or through low temperature fractional crystallization.

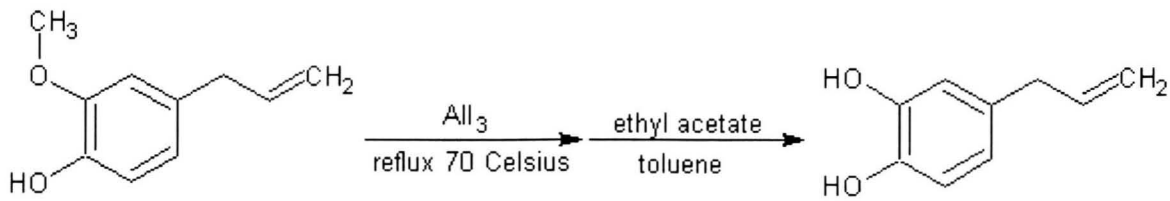
Hazards: Extinguish all flames and use care when handling hexane, diethyl ether, and ethyl acetate, all of which are highly flammable. Hexane and diethyl ether are capable of forming explosive mixtures with air. Wear gloves and use proper ventilation when handling iodine, which is capable of causing skin irritation and which is highly irritating to the nose and throat. Use care when handling sodium hydroxide and hydrogen chloride. Avoid inhalation of toluene vapors.

Procedure:

Personnel notes for procedure C: Safrole

Step 1: Preparation of 4-allylbenzene-1,2-diol (method 1)

Into a suitable reflux apparatus (equipped with motorized stirrer or other stirring means, and empty addition funnel), place 100 milliliters of dry hexane, followed by 2.7 grams of aluminum foil, and then followed by 38 grams of dry iodine. Immediately thereafter, reflux this entire mixture at about 70 Celsius for about 1 to 2 hours. During the reflux period, rapidly stir the reaction mixture. After refluxing for about 1 to 2 hours (the reaction mixture should have a gray color to it), remove the heat source, and allow the reaction mixture to cool to room temperature. Now, into the empty addition funnel on the reflux apparatus, place 16 grams of eugenol, followed by 900 milligrams of n-butyl ammonium iodide as catalyst, followed by 15 milliliters of dry hexane. Thereafter, slowly add drop-wise, the contents in the addition funnel, to the reaction mixture over a period sufficient to keep the reaction mixtures temperature below 40 Celsius. During the addition, rapidly stir the reaction mixture. After the addition, reflux the reaction mixture at 70 Celsius with rapid stirring for about 30 minutes. After refluxing for about 30 minutes, remove the heat source, and allow the reaction mixture to cool to room temperature. Thereafter, pour the entire reaction mixture into a suitable sized beaker, and then slowly add in 60 milliliters of ice water. Note: during the addition of the ice water, rapidly stir the reaction mixture in the beaker using any suitable stirring means. After the addition of the ice water, allow the entire mixture to stand for 1 hour. Thereafter, quickly filter-off the insoluble solids, and then quickly vacuum dry or air-dry these solids. Note: vacuum filtering and drying under inert atmosphere works best. Immediately after vacuum drying the solids, immerse these dry solids into 75 milliliters of dry ethyl acetate (contained in a suitable flask), and then swirl the flask for a few minutes. Then filter-off any insoluble impurities, and then place the ethyl acetate mixture into a distillation apparatus, and distill-off the ethyl acetate at 77 Celsius. Note: in some cases some water may be present with the ethyl acetate. This water can be separated from the ethyl acetate by use of a seperatory funnel—the water will be the lower layer, and can easily be drained-off. When no more ethyl acetate passes over or is collected, stop the distillation process, and recover the left over reaming residue (after it has cooled). Thereafter, recrystallize this collected left over residue from 75 milliliters of toluene, and after the recrystallization process, vacuum dry or air-dry the filtered-off crystals.



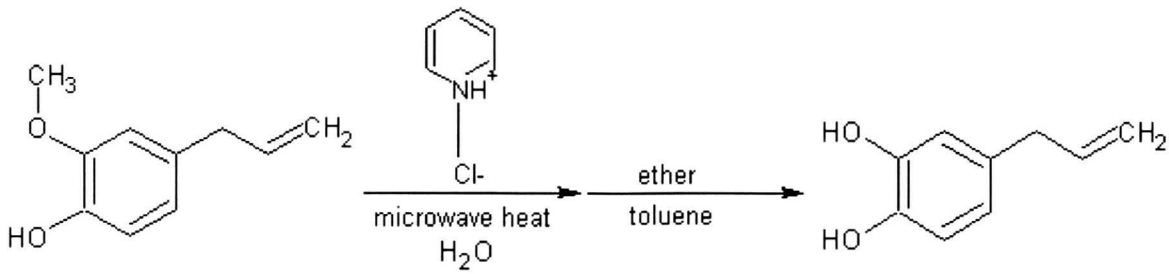
Eugenol

4-allylbenzene-1,2-diol

Step 1: Preparation of 4-allylbenzene-1,2-diol (method 2)

Into a suitable beaker, place 10 grams of pyridine, followed by 50 milliliters of diethyl ether, and then place this beaker into an ice bath. Thereafter, bubble into this pyridine/ether mixture, 5 grams of dry hydrogen chloride gas. After the addition of the hydrogen chloride gas, allow the entire mixture to stand at 0 Celsius for about 1 hour. Note: a freezer can be used rather then an ice bath. After 1 hour, filter-off the crystals of pyridine hydrochloride, and then place these crystals into a desiccator filled with anhydrous sodium sulfate until dry. Note: do not vacuum dry or air-dry the crystals as they will volatize away. When the crystals of the hydrochloride are dry, remove them from the desiccator, and place them into a suitable sized single neck flask. Then add in, 3 grams of eugenol, and then stopper the flask (preferably use a round bottom flask with a ground glass joint with glass stopper). Now, place this flask into a standard microwave, and microwave on medium (perhaps defrost, baked potato,

and/or sure simmer setting), for about 2 minutes. After 2 minutes, remove the flask from the microwave, and allow it to cool to room temperature. Thereafter, place the flask back into the microwave, and re-heat for another 2 minutes using the same heat setting. After 2 minutes, remove the flask once again, and allow it to cool to room temperature. Then repeat this process 5 more times, using the same heat period for 2 minutes each time. After the fifth and final heating time, remove the flask from the microwave, and allow it to cool to room temperature. Then add in 75-milliliters of ice water, and then sir the entire mixture for about 30 minutes. Thereafter, extract this entire mixture with three 50-millilter portions of diethyl ether. After the extraction process, combine all ether portions (if not already done so), and then dry this combined ether portion by adding to it, 15 grams of anhydrous sodium sulfate, and then stir the entire mixture for about 10 minutes—thereafter, filter-off the sodium sulfate. Finally, place this filtered ether portion into a distillation apparatus, and distill-off the ether at 40 Celsius. When no more ether passes over or is collected, stop the distillation process, and recover the left over remaining residue (after it has cooled). Then recrystallize this left over residue from 50 milliliters of toluene, and after the recrystallization process, vacuum dry or air-dry the filtered-off crystals.

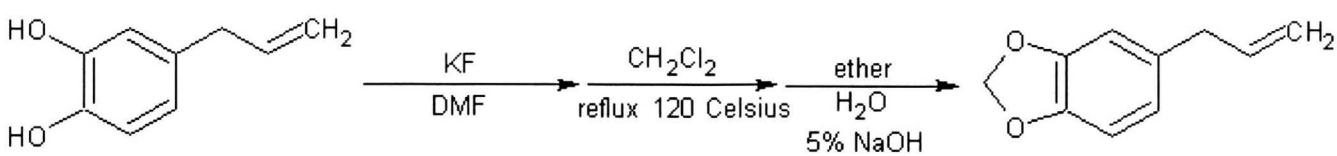


Eugenol

4-allylbenzene-1,2-diol

Step 2: Preparation of safrole

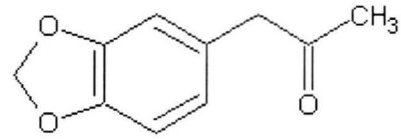
Into a suitable reflux apparatus, place 15 grams of the product obtained in step 1 (Note: step 1 should be repeated several times to produce enough 4-allylbenzene-1,2-diol intermediate to equal the necessary 15 grams—step 1, method 2 will have to be repeated several times to produce enough intermediate to equal about 15 grams). Immediately thereafter, add in 6 grams of potassium fluoride, followed by 100 milliliters of dimethylformamide (DMF), and then rapidly stir the entire reaction mixture for about 30 minutes at room temperature. After 30 minutes, add in 10 grams of methylene chloride, and then carefully reflux the entire reaction mixture at 120 Celsius for about 45 minutes. After refluxing for about 45 minutes, remove the heat source, and allow the reaction mixture to cool to room temperature. Thereafter, add in 50 milliliters of cold water, and then stir the entire mixture for about 10 minutes. Then extract the entire reaction mixture with three 50-milliliter portions of diethyl ether, and after the extraction process, combine all ether extracts (if not already done so), and then wash this combined ether portion with two 50-militer portions of ice cold water (to remove DMF), and then wash the combined ether portion with three 50-milliilter portions of a 5% sodium hydroxide solution. Note: after the extraction and washing portions, the ether will be the upper layer each time. After the extraction and washings, dry the diethyl ether portion by adding to it, 15 grams of anhydrous sodium sulfate, and then stir the entire mixture for about 10 minutes. Thereafter, filter-off the sodium sulfate, and then place the filtered ether portion into a distillation apparatus, and distill-off the ether at 40 Celsius. When no more ether passes over or is collected, stop the distillation process, and then recover the left over oily residue (after it has cooled), and then purify this oily residue by either vacuum distillation as in process A, or purify it by dissolving it into 75 milliliters of 95% ethyl alcohol, followed by filtration to remove insoluble impurities, and then distilling-off the ethyl alcohol. Note: other methods of purification may work other then vacuum distillation. Because vacuum distillation is rather expensive and impractical for most, try purifying the safrole from the oily left over residue by freezing it, as safrole has a melting point of around 11 Celsius, crystals of safrole should form at this cooler temperature, and they can then be filtered-off.



4-allylbenzene-1,2-diol

Safrole

Intermediate-0011. Piperonylacetone. 3,4-methylenedioxyphenylacetone. *1-(1,3-benzodioxol-5-yl)acetone*



Procedure A: Synthesis of piperonylacetone

Materials:

1. 200 grams of safrole (see Intermediate-0010. Safrole)	12. 15 grams of anhydrous magnesium sulfate
2. 500 grams of 99% ethyl alcohol	13. or 28.9 grams of potassium permanganate
3. or 30 grams of calcium oxide	14. or 29.8 grams of isosafrole
4. or 2 grams of potassium hydroxide	15. or 150 milliliters of diethyl ether
5. or 200 grams of safrole (see Intermediate-0010. Safrole)	16. or 15 grams of anhydrous magnesium sulfate
6. 28.9 grams of potassium permanganate	17. or 30 milliliters of methanol
7. 17 grams of 30% hydrogen peroxide	18. or 180 grams of a 15% sulfuric acid solution
8. 75 grams of 80% formic acid	19. or 150 milliliters of diethyl ether
9. 180 milliliters of a 15% sulfuric acid solution	20. or 75 milliliters of a 10% sodium carbonate solution
10. 150 milliliters of diethyl ether	21. or 15 grams of anhydrous magnesium sulfate
11. 150 milliliters of a 15% sodium hydroxide solution	

Summary: Piperonylacetone is prepared by first, rearranging safrole to isosafrole by either refluxing the safrole with potassium hydroxide and alcohol at moderate temperature, or by refluxing safrole with calcium oxide and potassium hydroxide at higher temperatures. The isosafrole is then collected after the unchanged safrole has been removed via distillation. The isosafrole is then converted into piperonylacetone by reaction with hydrogen peroxide in the presence of formic acid. The reaction mixture is then refluxed, treated with dilute sulfuric acid, and then extracted with ether to recover the desired product, which can be obtained upon removal of the ether. In a modified step 2, the isosafrole can be converted into the desired piperonylacetone by reaction with potassium permanganate. The reaction is kept at ice-cold temperatures to prevent unwanted side reactions. Thereafter, the reaction mixture is separated, treated with ether, and then refluxed with sulfuric acid and methanol. The desired product is then extracted with ether, and the ether then removed.

Hazards: Extinguish all flames before using diethyl ether, which is highly flammable and can form explosive mixtures with air. 99% ethyl alcohol, and methanol are both flammable, and methanol burns with an invisible flame, so keep out of contact with sources of ignition. Potassium hydroxide, calcium oxide, and concentrated formic acid can cause skin irritation, so wear gloves when handling. Potassium permanganate is a powerful oxidizing agent, so keep out of contact with combustible materials.

Procedure:

Personnel notes for procedure A: Piperonylacetone

Step 1: Preparation of isosafrole by rearrangement of safrole (method 1)

Into a suitable dry reflux apparatus, place 200 grams of safrole, followed by 500 grams of 99% ethyl alcohol, and then followed by 90 grams of potassium hydroxide. Immediately thereafter, reflux the entire mixture at 80 Celsius for 5 hours. After refluxing for about 5 hours, quickly replace the reflux condenser with a conventional condenser (fitted to a receiver flask), and then distill-off the ethyl alcohol. When no more ethyl alcohol passes over, stop the distillation process, and allow the remaining mixture to cool to room temperature. Thereafter, filter-off the insoluble potassium hydroxide, and then place the filtered mixture into a distillation apparatus, and distill the mixture at 234 Celsius to remove the unchanged safrole. Note: the ethyl alcohol, potassium hydroxide, and unchanged safrole can be recycled for additional isosafrole production. A vacuum distillation apparatus should be used to remove the safrole for more convenience. If a vacuum distillation apparatus is available, distill the mixture at 100 Celsius under a vacuum of 11 millimeters of mercury to obtain the safrole fraction. After

the safrole has been removed, recover the left over residue remaining (after it has cooled to room temperature), and then set it aside for step 2. Note: the conversion of safrole to isosafrole in this process is about 55%.

Step 1: Preparation of isosafrole by rearrangement of safrole (method 2)

In a modified process, place 30 grams of calcium oxide, followed by 2 grams of potassium hydroxide, followed by 200 grams of safrole into a suitable sized reflux apparatus, and then reflux the mixture at 234 Celsius for about 30 minutes. After the reflux period, remove the heat source, and allow the mixture to cool to room temperature. Thereafter, filter-off the calcium oxide and potassium hydroxide. Then place the filtered mixture into a distillation apparatus, and distill-off the unchanged safrole by heating to 234 Celsius, or use a high vacuum at 100 Celsius under 11 millimeters of mercury. When no more safrole is collected, stop the distillation process, and recover the left over remaining residue (after it has cooled to room temperature), and set aside for step 2. The conversion of safrole to isosafrole is 90%.

Step 2: Preparation of piperonylacetone (method 1)

Into a suitable 3-neck flask fitted with motorized stirrer, thermometer, and addition funnel, place 17 grams of 30% hydrogen peroxide, followed by 75 grams of 80% formic acid. Then briefly stir the entire mixture to form a uniform mixture. Now, prepare a solution by adding and dissolving 16.2 grams of isosafrole (prepared in step 1) into 60 milliliters of acetone. Then place this solution into the addition funnel, and then add it, drop-wise to the hydrogen peroxide/formic acid solution over a period sufficient to keep the reaction mixture below 40 Celsius at all times. During the addition, rapidly stir the reaction mixture. Note: a cold-water bath may or may not be needed to keep the reaction mixture below 40 Celsius. After the addition, continue to stir the reaction mixture for 8 hours at a temperature below 40 Celsius. After 8 hours, place the entire reaction mixture into a distillation apparatus, and remove the acetone solvent, and formic acid by heating to 101 Celsius. Note: distillation under reduced pressure works best. After the acetone and formic acid have been removed, stop the distillation process, and recover the left over remaining residue (after it has cooled to room temperature). Thereafter, place this residue into a clean large beaker, and then add in, 30 milliliters of methanol, followed by 180 milliliters of a 15% sulfuric acid solution. Then stir the entire mixture for about 30 minutes to form a uniform mixture. Thereafter, reflux this entire mixture at 100 Celsius for 90 minutes. After refluxing the mixture for about 90 minutes, stop the reflux process, and allow the mixture to cool to room temperature. Finally, extract the entire mixture with three 50-milliliter portions of diethyl ether, and after the extraction process, combine all ether portions (if not already done so), and then wash this combined ether portion with three 50-milliliter portions of cold water, and then wash the ether portion with three 50-milliliter portions of a 15% sodium hydroxide solution. Note: after the extraction, and washings, the ether will be the upper layer each time. After washing the ether portion with sodium carbonate solution, dry it by adding to it, 15 grams of anhydrous magnesium sulfate. Then stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Then place the filtered ether mixture into a distillation apparatus, and remove the ether. When no more ether passes over or is collected, recover the left over remaining residue (after it has cooled to room temperature), and then store the piperonylacetone in a cool dry place until use. Note: this recovered residue will contain predominately the desired piperonylacetone, which can be purified by vacuum distillation at 168 Celsius under a vacuum of 22 millimeters of mercury, if desired.

Step 2: Preparation of piperonylacetone (method 2)

Into a suitable beaker, place 28.9 grams of potassium permanganate, followed by 1400 milliliters of cold water. Thereafter, stir the mixture until all the potassium permanganate has dissolved (the solution will become dark purple). Thereafter, place 29.8 grams of isosafrole (prepared in step 1) into a suitable flask equipped with thermometer and addition funnel, and then place this flask and its contents into an ice bath, and chill the isosafrole to 0 Celsius. When the isosafrole reaches a temperature of 0 Celsius, place the potassium permanganate solution into the addition funnel, and then add it drop-wise, to the isosafrole over a period sufficient enough to keep the isosafrole reaction mixture below 5 Celsius at all times. During the addition of the potassium permanganate, rapidly stir the reaction mixture. Note: during the reaction, manganese dioxide will steadily precipitate, and the dark purple colored solution will fade. After the addition of the potassium permanganate solution, continue to stir the reaction mixture until the purple color of the solution nearly fades. When this happens, filter the entire reaction mixture to remove the insoluble manganese dioxide, and then place the entire filtered reaction mixture into a large seperatory funnel, and recover the upper organic layer (after removing the lower aqueous layer). Thereafter, extract this recovered upper organic layer with three 50-milliliter portions of diethyl ether, and after the extraction process, combine all ether portions (if not already done so), and then dry this combined ether portion by adding to it, 15 grams of anhydrous magnesium sulfate. Note: the recovered upper organic layer may entirely dissolve in the first portion of ether. If this is the case, add to it, two additional portions of ether (50 milliliters each). Note: if the organic layer does not all dissolve into the first portion of ether, recover the ether portion in the usual manner—as it will be the upper layer each time. After adding the magnesium sulfate to the ether mixture, stir the entire ether mixture for about 10 minutes—thereafter, filter-off the magnesium sulfate. Then place the filtered ether mixture into a distillation apparatus, and remove the ether. When no more ether passes over or is collected, remove the left over remaining residue (after it has cooled to room temperature), and then place it into a clean suitable sized

beaker. Now, add in 30 milliliters of methanol, followed by 180 grams of a 15% sulfuric acid solution. Then stir the entire mixture for about 30 minutes to form a uniform mixture. Thereafter, reflux this entire mixture at 70 Celsius for 90 minutes. After refluxing the mixture for about 90 minutes, stop the reflux process, and allow the mixture to cool to room temperature. Finally, extract the entire mixture with three 50-milliliter portions of diethyl ether, and after the extraction process, combine all ether portions (if not already done so), and then wash this combined ether portion with three 50-milliliter portions of cold water, and then wash the ether portion with three 25-milliliter portions of a 10% sodium carbonate solution. Note: after the extraction, and washings, the ether will be the upper layer each time. After washing the ether portion with sodium carbonate solution, dry it by adding to it, 15 grams of anhydrous magnesium sulfate. Then stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Then place the filtered ether mixture into a distillation apparatus, and remove the ether. When no more ether passes over or is collected, recover the left over remaining residue (after it has cooled to room temperature), and then store the piperonylacetone in a cool dry place until use. Note: this recovered residue will contain predominately the desired piperonylacetone, which can be purified by vacuum distillation at 168 Celsius under a vacuum of 22 millimeters of mercury, if desired.

Procedure B: Synthesis of piperonylacetone from black pepper

Materials:

1. 75 grams of powdered or finely ground black pepper	11. 375 milliliters of diethyl ether
2. 1075 milliliters of 95% ethyl alcohol	12. 200 milliliters of methylene chloride
3. 110 milliliters of a 10% potassium hydroxide solution in 95% ethyl alcohol	13. 7.2 grams of liquid bromine
4. 100 milliliters of acetone	14. 10 milliliters of methyl alcohol
5. 575 milliliters of 99% isopropyl alcohol	15. 1.4 grams of nitroethane
6. 100 grams of a 20% hydrochloric acid solution	16. 20 milliliters of a 10% hydrochloric acid solution
7. 100 milliliters of benzene	17. 4 grams of iron power
8. 10.4 grams of sodium hydroxide	18. 250 milligrams of ferric chloride hexahydrate
9. 4 grams of potassium permanganate	19. 14 milliliters of 35 to 38% hydrochloric acid
10. 30 milliliters of a 22% sodium bisulfite solution (saturated solution)	20. 15 grams of anhydrous sodium sulfate

Summary: Piperonylacetone can be prepared in an interesting fashion starting with the isolation of piperine from a very common food product. This common food product is merely regular black pepper, which is readily available, and relatively inexpensive. This piperine compound, is readily obtained by extraction of finely ground black pepper with either 95% ethyl alcohol, or 99% isopropyl alcohol. After the extraction, the alcohol extract is concentrated through evaporation, and then the desired product crystallized out, either by itself, or through the usual means. The isolated piperine is then converted into the intermediate piperic acid by refluxing with alcoholic potassium hydroxide in ethyl alcohol. After the reaction, the solvent is stripped, and the left over residue is then treated with water, boiled, and then acidified with hydrochloric acid. The acidified mixture is then allowed to cool, where upon the desired piperic acid precipitates, the piperic acid is then collected by filtration, dried, and then recrystallized in the usual manner from ethyl alcohol. The dried collected crystals after recrystallization are then converted into piperonal by either oxidization with potassium permanganate in benzene, or by reaction with bromine followed by sodium hydroxide. In both cases, the reaction mixture is then treated in the usual manner, and the desired product obtained. The desired product of piperonal is then converted into the nitro intermediate by condensation with nitroethane. The reaction is similar to the other nitro condensations found in this book, and the desired intermediate is then recovered by filtration. The dry nitro intermediate is then converted into the desired piperonylacetone by reduction with iron in the presence of a small amount of ferric chloride catalyst. The reaction mixture is then filtered, extracted with methylene chloride, acidified, and the desired product then obtained by separating the methylene chloride layer, drying, and then evaporating under the normal techniques.

Hazards: Extinguish all flames before using diethyl ether, which is highly flammable and capable of forming explosive mixtures with air. Extinguish all sources of ignition before using acetone, ethyl alcohol, isopropyl alcohol, benzene, methyl alcohol, and nitroethane. Nitroethane is highly flammable, so use caution. Methyl alcohol burns with a colorless flame, so burning alcohol cannot be seen. Iron powder is very flammable, and burns with excessive heat—avoid sources of ignition. Wear gloves when handling sodium hydroxide, concentrated hydrochloric acid, and liquid bromine. Use proper ventilation when handling liquid bromine, and avoid inhalation or skin contact. Liquid bromine is highly irritating to the eyes nose and throat, so use caution.

Procedure:

Personnel notes for procedure B: Piperonylacetone

Step 1: Extraction of piperine from black pepper (method 1)

Into a standard reflux apparatus, place 75 grams of powdered or finely ground black pepper (if using fresh black pepper corns or granules, the corns or granules should be finely ground before using). Note: 75 grams of black pepper is about 2/3 of a normal bottle sold in the grocery store. After adding the black pepper to the reflux apparatus, add in 750 milliliters of 95% ethyl alcohol. Thereafter, reflux the mixture at 78 Celsius for about 4 or 5 hours. After the reflux extraction process, remove the heat source, and allow the alcohol mixture to cool to room temperature. Thereafter, filter the alcohol extract to remove insoluble materials, and then place this filtered alcohol extract into a distillation apparatus, and distill-off the ethyl alcohol at 78 Celsius until the total remaining volume is about 75 milliliters. When most of the ethyl alcohol has been removed, and the left over remaining alcohol concentrate is around 75 milliliters in volume, stop the distillation process, and collect the left over remaining alcohol concentrate (after it has cooled), and place it into a clean beaker. Then, into a second clean beaker, add in 50 milliliters of a 10% potassium hydroxide solution in 95% ethyl alcohol. Thereafter, to the potassium hydroxide/alcohol solution, add in the concentrated alcohol extract, and thereafter, heat the total mixture at about 60 to 70 Celsius. When the temperature of this mixture reaches 60 to 70 Celsius, slowly add drop wise, 65 milliliters of warm water. Note: during the addition of the water, the desired piperine compound will gradually precipitate. When precipitation begins, remove the heat source, and allow the alcohol mixture to cool to room temperature, and during this cooling period continue to add the water, slowly and drop-wise. When the mixture has cooled to room temperature, add in 65 milliliters of more water (cold water this time), and then stir the entire mixture for about 30 minutes at room temperature, and then allow the entire mixture to stand (no stirring) for several hours at room temperature. Afterwards, filter-off the precipitated solid, and then vacuum dry or air-dry it. Finally, recrystallize this dry solid from 100 milliliters of acetone, and after the recrystallization process, vacuum dry or air-dry the filtered-off crystals. The result will be about 3 grams of the desired piperine compound with a melting point of 128 Celsius.

Step 1: Extraction of piperine from black pepper (method 2)

Into a standard reflux apparatus, place 75 grams of powdered or finely ground black pepper (if using fresh black pepper corns or granules, the corns or granules should be finely ground before using). Note: 75 grams of black pepper is about 2/3 of a normal bottle sold in the grocery store. After adding the black pepper to the reflux apparatus, add in 500 milliliters of isopropyl alcohol, followed by 5 grams of calcium carbonate. Thereafter, reflux the mixture at 81 Celsius for about 4 hours. After refluxing for about 4 hours, remove the heat source, and allow the alcohol mixture to cool to room temperature. Thereafter, filter the alcohol mixture to remove any insoluble materials, and then place the filtered alcohol mixture thereafter, into a distillation apparatus. Thereafter, distill-off the isopropyl alcohol at 81 Celsius but only to the point where 50 milliliters of total volume remains. When most of the isopropyl alcohol has been removed, stop the distillation process, and recover the left over remaining alcohol concentrate (before it cools), and then place this alcohol concentrate into a clean beaker, and allow it to cool. After it has cooled to room temperature, place it into an ice bath, and chill it to about 0 Celsius. Then let the alcohol concentrate stand at 0 Celsius for about 1 hour. After sitting for 1 hour, filter the alcohol concentrate to recover the precipitated solid product. Thereafter, vacuum dry or air-dry this filtered-off product. Finally, recrystallize the filtered-off product from 100 milliliters of acetone, and after the recrystallization process, vacuum dry or air-dry the filtered-off crystals.

Step 2: Preparation of piperic acid

Note: perform step 1 two more times to acquire at least 8 grams of piperine. Then place 6 grams of piperine (obtained in step 1) into a reflux apparatus, and then add in 60 milliliters of a 10% potassium hydroxide solution in 95% ethyl alcohol. Thereafter, reflux this mixture at 78 Celsius for 4 hours. After refluxing for about 4 hours, quickly replace the reflux condenser with a standard condenser (fitted with a receiver flask), and then distill-off the ethyl alcohol at 78 Celsius until no more ethyl alcohol passes over or is collected. When no more ethyl alcohol is removed, stop the distillation process, and then recover the left over remaining residue (after it has cooled). Note: the ethyl alcohol distillate contained in the receiver flask will contain dissolved Piperidine. Then place this left over recovered residue into a clean beaker, and then add in 300 milliliters of water. Then gently heat this mixture to about 100 Celsius with stirring (using a motorized stirrer or magnetic stirrer bar), and when its temperature reaches about 100 Celsius, add in 100 grams of a 20% hydrochloric acid solution. After the addition of the hydrochloric acid solution, continue to stir the mixture at 100 Celsius for about 30 minutes. After 30 minutes, remove the heat source, and then filter the mixture before it cools to recover the precipitated product of piperic acid. Then vacuum dry or air-dry the filtered-off product. Note: additional product may be obtained by allowing the filtered mixture to cool to room temperature and then allowing it to stand at room temperature for about 1 hour. If after doing this, you notice additional precipitation (in this case the precipitation of a yellowish solid), go ahead and filter it off, and add it to the other filtered-off crop. If however, the precipitate

is not yellow, don't bother. All the filtered-off desired product should be vacuum dried or air-dried, and then recrystallized from about 300 milliliters of boiling 95% ethyl alcohol.

Step 3: Preparation of piperonal (method 1)

Into a suitable flask equipped with motorized stirrer, thermometer, and addition funnel, place 5 grams of piperic acid (obtained in step 2), followed by 100 milliliters of benzene, and then followed by 2 grams of sodium hydroxide. Thereafter, chill this mixture to about 0 Celsius, and then prepare a solution by adding and dissolving 4 grams of potassium permanganate into 60 milliliters of water. Thereafter, place this potassium permanganate solution into the addition funnel, and then slowly add this potassium permanganate solution, drop-wise, over a period sufficient to keep the reaction mixtures temperature below 5 Celsius at all times. During the addition of the potassium permanganate, rapidly stir the reaction mixture. After the addition of the potassium permanganate solution, rapidly stir the reaction mixture at a temperature below 5 Celsius for about 90 minutes. After 90 minutes, gradually add to the reaction mixture, 30 milliliters of a 22% sodium bisulfite solution (saturated solution), and then rapidly stir the reaction mixture for 30 minutes at a temperature below 5 celsius at all times. Note: during the addition of the sodium bisulfite solution, rapidly stir the reaction mixture and watch out for heat build-up as excess permanganate destroys the sulfite. After the addition of the sodium sulfite, and after stirring the mixture for about 30 minutes, filter-off the precipitated impurities, and then wash the filtered-off precipitated impurities with three 25-mililiter portions of diethyl ether, several times using the same washing portions (to dissolve any product that may have been filtered-off—combine all three ether washing portions once finished). Thereafter, extract the filtered reaction mixture with three 25-milliliter portions of diethyl ether, and after the extraction process, combine all ether portions (if not already done so), and then combine this combined ether portion with the previous ether washing portions. Note: during all extraction and washings, the ether will be the upper layer each time. Then place the total combined ether portion into a distillation apparatus, and distil-off the ether. When no more ether passes over or is colleted, recover the left over remaining residue (after it has cooled), and then recrystallize this residue from 75 milliliters of fresh diethyl ether. After the recrystallization process, vacuum dry or air-dry any filtered-off solids.

Step 3: Preparation of piperonal (method 2)

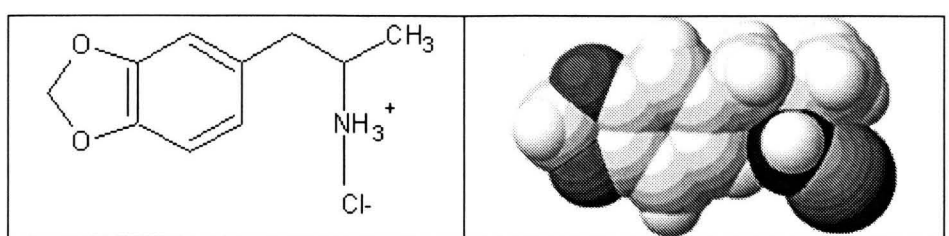
Into a suitable beaker or flask, equipped with motorized stirrer or magnetic stirrer, and thermometer, place 5 grams of piperic acid (prepared in step 2), followed by 75 milliliters of methylene chloride. Thereafter, place this mixture into an ice water bath, and chill the mixture to about 5 Celsius. Then prepare a bromine solution by adding and dissolving 7.2 grams of liquid bromine into 75 milliliters of methylene chloride. Then gradually add this bromine/methylene chloride mixture to the piperic acid mixture over a period of time sufficient to keep the reaction mixture at a temperature below 10 Celsius. During the addition, rapidly stir the reaction mixture. After the addition, rapidly stir the reaction at a temperature below 10 Celsius for about 1 hour. Thereafter, remove the ice water bath, and allow the reaction mixture to warm to room temperature. Then, place this warmed reaction mixture into a suitable reflux apparatus, fitted with an addition funnel and motorized stirrer, and then gently reflux at about 40 Celsius. Thereafter, place a hot sodium hydroxide solution (around 55 to 65 Celsius) into the addition funnel, and then add to the reaction mixture, drop-wise, this hot sodium hydroxide solution over a period of about 5 or 10 minutes while rapidly stirring the reaction mixture. This sodium hydroxide solution is prepared by adding and dissolving 8.4 grams of sodium hydroxide into 50 milliliters of water. Note: the addition of sodium hydroxide to water generates much heat, but this addition may not produce enough heat to reach the desired 55 to 65 Celsius range. If after dissolving the sodium hydroxide into water, the solutions temperature is below the recommended temperature, simply heat the mixture to the desired temperature before using. During the addition of the sodium hydroxide, rapidly stir the reaction mixture. After the addition of the hot sodium hydroxide solution, continue to reflux the entire reaction mixture at about 40 Celsius for about 1 hour. During this 1-hour reflux period, rapidly stir the reaction mixture. After refluxing for about 1 hour, remove the heat source, and allow the reaction mixture to cool to room temperature. Thereafter, filter the reaction mixture to remove any potential insoluble materials, and then wash the filtered-off materials with three 25-millilter portions of diethyl ether (to dissolve any desired product that may have been filtered-off—after the extraction process, combine all the ether portions and set aside for a short period). Note: this filtering and washing process may not be needed. Afterwards, place the reaction mixture into a seperatory funnel, and then recover the lower methylene chloride layer. The upper aqueous layer can be discarded or recycled if desired—will contain dissolved sodium bromide, which can be recovered and recycled for liquid bromine production. Then place the recovered lower methylene chloride layer into a distillation apparatus, and distill-off the methylene chloride at 40 Celsius. When no more methylene chloride passes over or is collected, stop the distillation process, and then recover the left over remaining residue (after it has cooled). Now, dissolve the bulk of the residue into 75 milliliters of diethyl ether, and then quickly filter this entire mixture to remove any potential insoluble materials. Then combine this filtered ether mixture with the previous ether washing portion (if there where any), and then place this ether mixture into a distillation apparatus, and distill-off the ether. When no more ether passes over or is collected, stop the distillation process, and recover the left over remaining residue (after it has cooled). Finally, recrystallize the recovered left over residue from 75 milliliters of diethyl ether, and after the recrystallization process, vacuum dry or air-dry the filtered-off crystals.

Step 4: Preparation of piperonylacetone

Into a suitable flask, equipped with motorized stirrer or magnetic stirrer, place 3 grams of the product obtained in step 3, followed by 10 milliliters of methyl alcohol. Then stir the entire mixture to dissolve the piperonal. Then add to piperonal mixture, 1.4 grams of nitroethane over a period sufficient to keep the reaction mixtures temperature around room temperature. During the addition, rapidly stir the reaction mixture. Thereafter, place this mixture into an ice bath, and chill to about 0 Celsius. When the reaction mixture reaches a temperature of about 0 Celsius, add in a ice cold sodium hydroxide solution prepared by adding and dissolving 1 gram of sodium hydroxide into 5 milliliters of water. Note: sodium hydroxide generates excessive heat when dissolved in water, so allow the solution to cool to room temperature before chilling it in a refrigerator (to avoid glass breakage). During the addition of the sodium hydroxide solution, rapidly stir the reaction mixture and keep its temperature below 5 Celsius at all times. After the addition, continue to rapidly stir the reaction mixture at a temperature below 5 Celsius for about 15 minutes. After stirring for about 15 minutes, add in 20 milliliters of water, and then stir the entire reaction mixture for about 30 minutes. Thereafter, carefully pour this entire diluted reaction mixture into 20 milliliters of a 10% hydrochloric acid solution (pre-chilled in a refrigerator to about 10 Celsius), which is contained in a suitable beaker. During the addition of the reaction mixture to the dilute hydrochloric acid solution, rapidly stir the dilute hydrochloric acid solution. After the addition, rapidly stir the acidified mixture for 30 minutes. Then, place the entire acidified mixture into an ice bath, and chill to about 0 Celsius. Then allow the mixture to stand at 0 Celsius for 1 hour. Thereafter, filter-off the precipitated product, and vacuum dry or air-dry. Finally, recrystallize this dried solid from 75 milliliters of 99% isopropyl alcohol, and after the extraction process, vacuum dry or air-dry the crystals to obtain the dry nitro intermediate.

Into a suitable 3-neck flask equipped with motorized stirrer, reflux condenser, and thermometer, place 3.3 grams of the product obtained in step 3, followed by 25 milliliters of 95% ethyl alcohol. Thereby, bring the mixture to reflux at 78 Celsius with stirring. When all solids dissolve, immediately thereafter, add in 60 milliliters of hot water (previously heated to about 60 celsius). Then add in small portions at a time, 4 grams of iron powder (through the top the reflux condenser) followed immediately by 250 milligrams of ferric chloride hexahydrate. Both additions should take no more then 15 minutes. Note: during both additions, rapidly stir the reaction mixture and maintain its temperature at reflux at 78 Celsius. After the addition, immediately add in 4 milliliters of 35 to 38% hydrochloric acid (muriatic acid of 31% will work) over a period of about 10 minutes. After the addition of the acid, continue to rapidly stir the reaction mixture for 30 minutes at reflux at 78 Celsius. Thereafter, quickly replace the reflux condenser with a standard condenser (fitted with receiver flask), and then distil-off the ethyl alcohol at 78 Celsius until no more alcohol passes over or is collected. When the ethyl alcohol has been removed, stop the distillation process, and recover the left over remaining contents (after it has cooled), and then filter this cooled left over mixture to remove any insoluble materials. Then quickly wash the filtered-off solids with three 10-milliliter portions of methylene chloride, several times using the same washing portion, and after the washing, combine all methylene chloride portions, and then add this combined methylene chloride portion to the filtered reaction mixture. Thereafter, add to this two-phase reaction mixture, 10 milliliters of 35 to 38% hydrochloric acid, and then rapidly stir the entire acidified reaction mixture for about 30 minutes. Finally, place the two-phase reaction mixture into a seperatory funnel, and remove the lower methylene chloride portion. Now, to the upper water layer, extract this upper water layer with two 10-millilter portions of methylene chloride, and after the extraction process, combine all methylene chloride portions (if not already done so), and then combine this combined methylene chloride portion with the previous methylene chloride portion. Finally, dry this total combined methylene chloride portion by adding to it, 15 grams of anhydrous sodium sulfate, and then stir the entire mixture for about 10 minutes—thereafter, filter-off the sodium sulfate. Then place this filtered methylene chloride portion into a distillation apparatus, and distill-off the methylene chloride at 40 Celsius. When no more methylene chloride passes over or is collected, recover the left over remaining oily residue (after it has cooled), and then place this desired product of piperonylacetone in a cool place until use.

0012. MDA hydrochloride. *1-(1,3-benzodioxol-5-yl)propan-2-amine hydrochloride*



Structure	3D model

MDA hydrochloride is a psychedelic drug with stimulation properties, however its stimulation properties are rather muffled by its strong intoxicating effects. The drug produces mild hallucinations upon administration, with secondary effects resulting in

bursts of energy, and/or feelings of well-being. MDA hydrochloride produces an interesting high when taken by users, and it has been explained by some to produce a "pleasant trip".

Note: This substance is a controlled substance (hallucinogen/stimulant) as listed in the US code of Federal regulations.

Toxicity: Low	**Rate of onset (average):** Rapid
Stimulation dosage (ingestion): 85 to 160 milligrams	**Duration of effects (average):** 8 to 12 hours (depending on the person)
Stimulation dosage (inhalation): 50 to 100 milligrams	**Habit forming potential:** Moderate
Stimulation dosage (injection): 30 milligrams +	**Estimated value U.S. (based on procedure):** $24 per gram

Procedure A: Preparation of MDA hydrochloride

Materials:

1. 90 grams of 47% hydrobromic acid	9. 20 milliliters of diethyl ether
2. 16 grams of safrole (see Intermediate-0010. Safrole)	10. 50 grams of calcium bicarbonate
3. 21 grams of dry hydrogen bromide gas	11. 20 grams of hexamine
4. 450 milliliters of diethyl ether	12. 31.5 grams of sodium iodide
5. 25 grams of anhydrous potassium carbonate	13. 40 grams of dry hydrogen chloride gas
6. or 40 milliliters of glacial acetic acid	14. 10 grams of sodium hydroxide
7. or 40 milliliters of 47% hydrobromic acid	15. 15 grams of anhydrous magnesium sulfate
8. or 20 grams of safrole (see Intermediate-0010. Safrole)	

Summary: MDA is prepared in a two-step process starting with the formation of bromosafrole. Bromosafrole can be prepared in several ways, but generally includes the bromination of the safrole with hydrobromic acid. The resulting bromosafrole is then obtained by solvent extraction, followed by removal of the solvent. The resulting bromosafrole is then converted into MDA by reaction with hexamethylenetetramine (hexamine) in the presence of sodium iodide and 95% ethyl alcohol. The reaction takes about 30 days, which is an astronomical amount, but little has to be done during this time period, as the reaction simply sits for the necessary amount of time. After 30 days, the reaction mixture is acidified with hydrogen chloride, and then treated with sodium hydroxide to liberate the freebase MDA. The freebase MDA is then extracted into ether, and the resulting ether mixture is then treated with hydrogen chloride gas to precipitate the MDA hydrochloride as the desired product.

Hazards: Use great care when handling concentrated hydrobromic acid, which is very irritating to the nose, and throat. Use proper ventilation when using hydrogen bromide, and hydrogen chloride gas. Extinguish all flames before using diethyl ether, which is highly flammable and can form explosive mixtures with air. Wear gloves when handling glacial acetic acid, and sodium hydroxide, as they both can cause skin irritation.

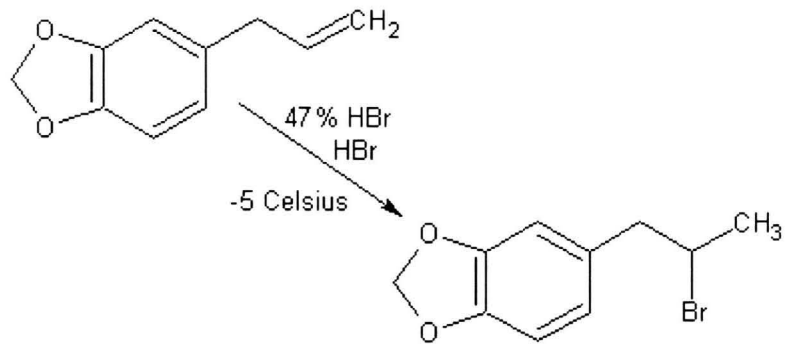
Procedure:

Personnel notes for procedure A: MDA hydrochloride

Step 1: Bromination of safrole (method 1)

Into a suitable flask equipped with motorized stirrer, thermometer, and addition funnel, place 90 grams of 47% hydrobromic acid, and then place 16 grams of safrole into the addition funnel. Thereafter, place the flask into an ice/salt bath, and chill to –5 Celsius. When the hydrobromic acid reaches a temperature of –5 Celsius, slowly add the safrole to the hydrobromic acid over a period sufficient to keep the reaction temperature below 0 Celsius at all times. During the addition, moderately stir the reaction mixture. Note: do not allow the reaction mixture to get above 0 Celsius, as unwanted side reactions will result decreasing the yield of desired product. After the addition of the safrole to the hydrobromic acid, replace the addition funnel with a gas inlet tube (make sure you leave a vent open to the atmosphere), and then bubble into the reaction mixture 21 grams of dry hydrogen bromide gas. During the addition of the hydrogen bromide gas, stir the reaction mixture, and maintain the temperature below 0 Celsius at all times. After the addition of the hydrogen bromide gas, continue to stir the reaction mixture at a temperature below 0 Celsius for 24 hours. Note: it may be possible to store the reaction mixture in a freezer at –5 Celsius instead of maintaining a pesky ice/salt bath, which has to be continuously replaced. If using a freezer, attach a sodium carbonate trap to the reaction flask, to capture any possible escaping acidic vapors. After allowing the reaction mixture to sit for 24 hours, remove the ice/salt bath, or take the reaction apparatus out of the freezer, and then pour the entire reaction mixture over 250

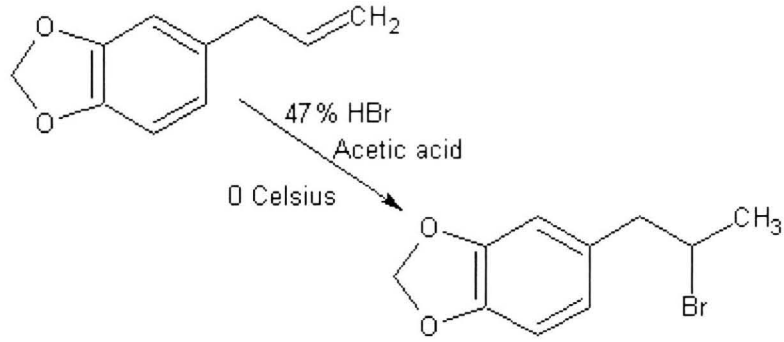
grams of crushed ice contained in suitable beaker. After the ice has melted, quickly extract the entire mixture before it gets warm, with three 75-milliliter portions of diethyl ether. After the extraction process, combine all ether portions if not already done so, and then dry the combined ether portion by adding to it, 25 grams of anhydrous potassium carbonate (also to remove any acidic agents). After the addition of the potassium carbonate, stir the entire ether mixture for about 15 minutes, and then filter-off any insoluble materials. Finally, place the filtered ether mixture into a distillation apparatus or rotary evaporator, and remove the ether. When no more ether passes over, remove the remaining left over residue (after it has cooled to room temperature), and then place it aside for step 2. The resulting bromosafrole can be purified by vacuum distillation at 148 Celsius under a vacuum of 14 millimeters of mercury if desired, but this is not generally needed for step 2.



Bromosafrole

Step 1: Bromination of safrole (method 2)

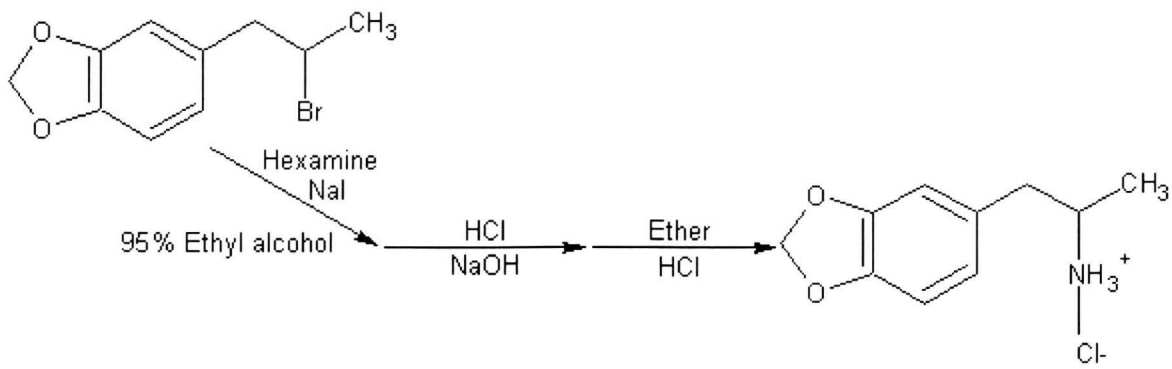
Into a suitable flask equipped with motorized stirrer, thermometer, and addition funnel, place 40 milliliters of glacial acetic acid, and then place 40 milliliters of 47% hydrobromic acid into the addition funnel (note: pre chill this hydrobromic acid to 0 Celsius before adding it to the addition funnel—a freezer can be used for this chilling process). Then place the flask into an ice bath, and chill to 0 Celsius. When the glacial acetic acid reaches a temperature of 0 Celsius, slowly add drop-wise, the pre chilled hydrobromic acid to the cooled glacial acetic acid, over a period sufficient enough to maintain the temperature of the glacial acetic acid below 5 Celsius at all times. Note: during the addition of the hydrobromic acid, moderately stir the reaction mixture. After the addition of the hydrobromic acid, replace the now empty addition funnel with a clean one, and then place 20 grams of safrole into it. Now, slowly add this safrole to the reaction mixture over a period sufficient to maintain the reaction mixture below 5 Celsius at all times. Note: during the addition of the safrole, rapidly stir the reaction mixture. After the addition of the safrole, allow the reaction mixture to sit for 24 hours at a temperature below 5 Celsius. During this sitting process, slowly stir the reaction mixture. Note: a refrigerator or freezer cooled to 0 Celsius can be used for the 24 hour sitting process. After 24 hours, remove the reaction mixture, and pour it into a suitable beaker, and then allow it to warm to room temperature over a period of several hours. Then, after letting the reaction mixture sit for a few hours, add to it, 100 grams of crushed ice, and then slowly stir the mixture until the ice melts. Once the ice has melted, pour the entire reaction mixture into a seperatory funnel, and then recover the red liquid layer. Note: this red liquid layer will contain the bromosafrole, and is usually the lower layer. Second note: in some cases, the entire reaction mixture may have a red color to it, making the phase boundary between the two layers difficult to see. If this is the case, hold the seperatory funnel up to a light to try and get a better view of where the phase boundary is. Once the red bromosafrole layer has been recovered, briefly extract the water layer with 20 milliliters of diethyl ether, and then recover this ether layer by using a seperatory funnel—it should be the upper layer. Then add this ether layer to the main red bromosafrole layer, and then wash this total combined mixture by adding to it, 50 grams of calcium bicarbonate, and then stir the entire mixture for10 minutes—thereafter, filter-off any insoluble materials. Finally, place this filtered mixture into a distillation apparatus, and remove the ether. Once the ether has been removed, recover the left over remaining product (after it has cooled), and then set it aside for step 2. Purification of this bromosafrole is not generally needed for step 2.



Bromosafrole

Step 2: Preparation of MDA

Into a suitable 3-neck flask equipped with motorized stirrer, and thermometer, place 20 grams of hexamine, followed by 31.5 grams of sodium iodide, and then add in 200 milliliters of 95% ethyl alcohol. Then stir the entire mixture for about 10 minutes, and then add in 48.6 grams of bromosafrole (obtained in step 1). Now, place the entire apparatus aside, and let it stand for 30 days at room temperature. During this 30-day period, slowly stir the reaction mixture. After 30 days, pour the entire reaction mixture into a clean suitable sized beaker, and then bubble into this reaction mixture, 20 grams of dry hydrogen chloride gas. During the addition of the hydrogen chloride, moderately stir the reaction mixture. After the addition of the hydrogen chloride, moderately stir the reaction mixture for 1 hour at ambient temperature (room temperature). After stirring for 1 hour, filter the reaction mixture to remove any insoluble materials (mainly ammonium chloride), and then place the filtered reaction mixture into a distillation apparatus or rotary evaporator, and distill-off the ethyl alcohol. Note: distilling-off the ethyl alcohol under vacuum works best. Once the ethyl alcohol has been removed, recover the left over remaining residue (after it has cooled), and then place it into a clean beaker. Thereafter, prepare a sodium hydroxide solution by adding and dissolving 10 grams of sodium hydroxide into 30 milliliters of water, and then allow this sodium hydroxide solution to cool before using it. Note: sodium hydroxide generates much heat when dissolved in water, so allow the solution to cool before using. Then add this sodium hydroxide solution to the left over remaining residue in the beaker, and then stir the whole basic mixture for about 1 hour at room temperature. Finally, extract this mixture with three 75-milliliter portions of diethyl ether, and after the extraction process, combine all ether portions (if not already done so), and then dry this combined ether portion by adding to it, 15 grams of anhydrous magnesium sulfate. Then sir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Now, place this ether mixture into an ice bath, and chill to 0 Celsius. Thereafter, bubble into this ether mixture, 20 grams (an excess) of dry hydrogen chloride gas. During the addition of the hydrogen chloride, moderately stir the ether mixture. After the addition of the hydrogen chloride, continue to stir the ether mixture for about 30 minutes, and then filter-off the precipitated MDA hydrochloride product. Then vacuum dry or air-dry the crystals.



MDA hydrochloride

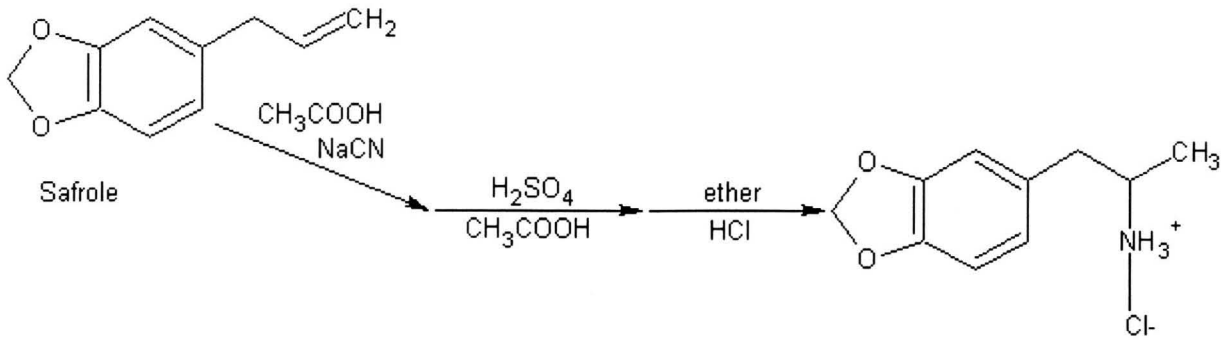
Note: Other salts of the freebase MDA such as the sulfate, tartrate, citrate, and phosphate can be prepared by adding the corresponding acid to the ether mixture of the MDA freebase compound obtained at the end of step 2. For sulfuric acid or tartaric acid, 1 mole of 98% sulfuric acid or d-tartaric acid should be added to 2 moles of the ether mixture of the MDA freebase. For citric acid or phosphoric acid, 1 mole of the acid should be added to 3 moles of the ether mixture of the MDA freebase. The ether mixture after treatment with the corresponding acid in each of these cases can then be filtered to recover the precipitated crystals of the desired product. All the salts of MDA are stimulants and are psychedelic in nature. The tartrate and citrate salts may be twice as potent as the hydrochloride or sulfate.

Procedure B: Preparation of MDA hydrochloride directly from safrole

Materials:

1. 100 milliliters of glacial acetic	5. 240 grams of sodium hydroxide
2. 68.4 grams of safrole (see Intermediate-0010. Safrole)	6. 300 milliliters of diethyl ether
3. 22 grams of sodium cyanide (technical grade 90%+)	7. 25 grams of anhydrous magnesium sulfate
4. 100 grams of 98% sulfuric acid	8. 20 grams of dry hydrogen chloride gas

Summary: MDA hydrochloride is readily prepared in a direct process starting with safrole. The safrole can be directly animated by reaction with sodium cyanide in the presence of sulfuric acid and glacial acetic acid. The reaction is generally mild, and afterwards, the acidic reaction mixture is neutralized by the addition of sodium hydroxide, and the resulting reaction mixture is then refluxed for a prolonged period of time, and then extracted with ether in the usual manner. The ether mixture thus containing the freebase of MDA is then treated with hydrogen chloride, whereby the MDA precipitates as the hydrochloride.



MDA hydrochloride

Hazards: Use proper ventilation when using hydrogen chloride gas, which is very irritating to the nose and throat. Extinguish all flames before using diethyl ether, which is highly flammable and can form explosive mixtures with air. Wear gloves when handling glacial acetic acid, sodium hydroxide, and sulfuric acid, as they can produce skin irritation. Note: this procedure involves the generation of hydrogen cyanide. Use maximum ventilation, and observe all precautions related to the use of hydrogen cyanide. Use an apparatus that includes a sodium hydroxide trap (as indicated in figure 003) to remove any cyanide vapors.

Procedure:

Personnel notes for procedure B: MDA hydrochloride

Into a suitable 3-neck flask (say about a 1500 ml) equipped with thermometer, motorized stirrer, and addition funnel, (see figure 003), place 50 milliliters of glacial acetic acid, followed by 68.4 grams of safrole, followed by 22 grams of sodium cyanide (technical grade 90%+). Thereafter, add to the addition funnel 100 grams of concentrated sulfuric acid, followed by 50 milliliters of glacial acetic acid. Then add this sulfuric acid/glacial acetic acid mixture drop-wise, to the safrole mixture over a period of about 1 hour. During the addition, rapidly stir the reaction mixture and maintain its temperature around room temperature. Note: a cold water bath may or may not be needed). After the addition of the sulfuric acid/glacial acetic acid, quickly replace the empty addition funnel with a clean one, and then prepare a sodium hydroxide solution by adding and dissolving 240 grams of sodium hydroxide into 500 milliliters of water, and then add this alkaline solution to a the new and clean addition funnel just attached. Note: sodium hydroxide generates much heat when added to water, so allow the sodium hydroxide solution to cool to room temperature before using. Now, add this sodium hydroxide solution, drop-wise, over a period sufficient to keep the reaction from over heating (keep the reaction mixture below 60 Celsius, as heat is generated by the addition of the sodium hydroxide—neutralizes the acids generating heat). During the addition of the sodium hydroxide solution, rapidly stir the reaction mixture. Thereafter, replace the second addition funnel with a reflux condenser, and then reflux the entire reaction mixture at about 90 to 100 Celsius for about 10 hours. During the reflux period, moderately stir the

reaction mixture. After heating for 8 hours, remove the heat source, and allow the reaction mixture to cool to room temperature. Finally, pour the entire reaction mixture into a clean seperatory funnel, and then extract it with three 100-milliliter portions of diethyl ether. Note: during the extraction process, the ether will be the upper layer each time. After the extraction period, combine all ether portions (if not already done so), and then wash this combined ether portion three times with 100 milliliters of cold water each time. Note: after the washings, the ether can be recovered using a seperatory funnel, as it will be the upper layer each time. Then dry this washed ether portion by adding to it, 25 grams of anhydrous magnesium sulfate. Then stir the entire ether mixture for about 10 minutes and then filter-off the magnesium sulfate. Thereafter, place the filtered ether mixture into an ice bath, and chill to 0 Celsius. Then bubble into this chilled ether mixture, 20 grams of dry hydrogen chloride gas (an excess). After the addition of the dry hydrogen chloride gas, allow the reaction mixture to stand at 0 Celsius for about 1 hour, and then filter-off the precipitated product of MDA hydrochloride, and then vacuum dry or air-dry these crystals.

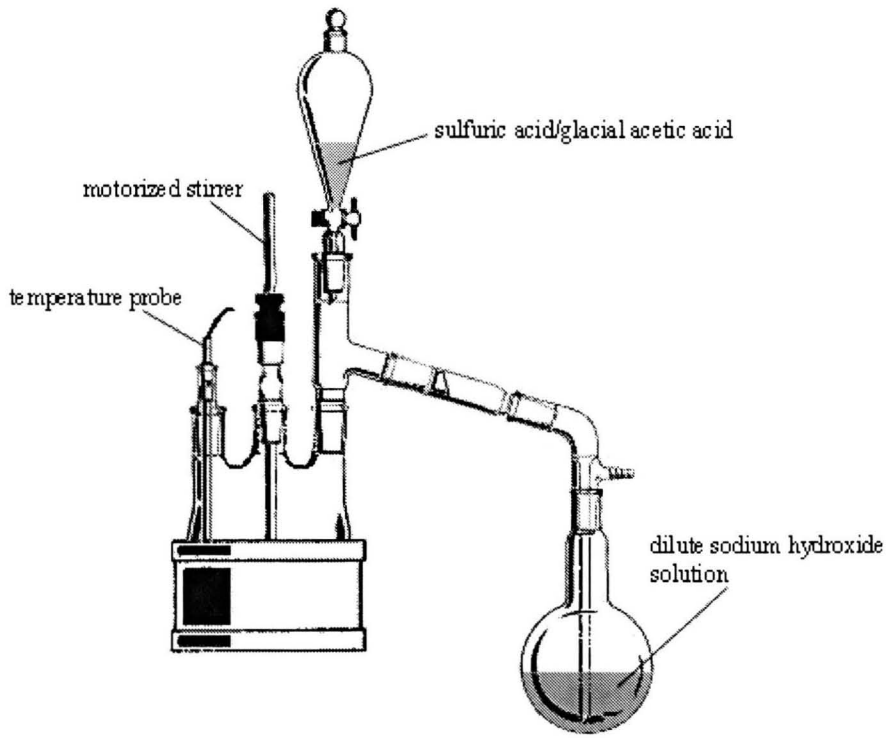


Figure 043. Apparatus for the addition of sulfuric acid to the sodium cyanide to generate hydrogen cyanide. Note: the sodium hydroxide trap is to neutralize any cyanide vapors. Second note: this apparatus design is only suggestive, and does not represent the actual necessary design—apparatus design may vary considerably.

Note: Other salts of the freebase MDA such as the sulfate, tartrate, citrate, and phosphate can be prepared by adding the corresponding acid to the ether mixture of the MDA freebase compound obtained at the end of the procedure. For sulfuric acid or tartaric acid, 1 mole of 98% sulfuric acid or d-tartaric acid should be added to 2 moles of the ether mixture of the MDA freebase. For citric acid or phosphoric acid, 1 mole of the acid should be added to 3 moles of the ether mixture of the MDA freebase. The ether mixture after treatment with the corresponding acid in each of these cases can then be filtered to recover the precipitated crystals of the desired product. All the salts of MDA are stimulants and are psychedelic in nature. The tartrate and citrate salts may be twice as potent as the hydrochloride or sulfate.

Procedure C: Preparation of MDA hydrochloride from bromosafrole using pressure apparatus

Materials:

1. 17 grams of dry ammonia gas	5. 300 milliliters of diethyl ether
2. 150 milliliters of methanol	6. 60 grams of sodium hydroxide
3. 24.5 grams of bromosafrole (see 0012. MDA hydrochloride, procedure A, step 1)	7. 15 grams of anhydrous magnesium sulfate
4. 60 grams of 35 to 38% hydrochloric acid	8. 15 grams of dry hydrogen chloride gas

Summary: MDA hydrochloride can be prepared from bromosafrole by ammonolysis using ammonia. The ammonia is initially dissolved in methanol, and the bromosafrole is then added. The reaction only takes place under heat and pressure, and a special design to the apparatus is employed. In this case, a regular party balloon can be used to create a pressurized environment

without the hazards associated with conventional pressure vessels. The reaction mixture is heated under this pressure for a prolonged period of time, whereby the MDA is formed. The MDA is then collected by first, acidifying the reaction mixture, and then treating the reaction mixture with ether to remove impurities, and the resulting acidified reaction mixture, after removal of the ether extracts, is then basified to liberate the MDA freebase. The freebase oil is then extracted into ether, and the resulting ether mixture is then treated with hydrogen chloride to precipitate the desired MDA hydrochloride.

Hazards: Use caution when handling ammonia, which is highly irritating to the nose and throat—use proper ventilation. Use proper ventilation when using hydrogen chloride gas, which is very irritating to the nose and throat. Extinguish all flames before using diethyl ether, which is highly flammable and can form explosive mixtures with air. Wear gloves when handling sodium hydroxide, and concentrated hydrochloric acid, as they can produce skin irritation

Procedure:

Personnel notes for procedure C: MDA hydrochloride

First, prepare a methanol/ammonia solution by bubbling 17 grams of dry ammonia gas into 150 milliliters of methanol. Thereafter, place this methanol/ammonia solution into a suitable sized flask, and then add in 24.5 grams of bromosafrole (prepared in procedure A of 0011). Thereafter, place a suitable sized balloon over the flask, and secure the balloon to the flask using a metal ring clamp. Then heat the contents in the flask to about 130 Celsius for 8 hours. Note: the balloon will inflate and deflate sporadically during the heating process. The balloon is deigned to keep the contents of the flask under pressure to properly carryout the reaction. If during the heating process, the balloon pops or explodes, quickly replace with another one, and continue the operation for the necessary amount of remaining time. After the necessary amount of time has been consumed, stop the heating process, and allow the reaction mixture in the flask to cool to room temperature. Note: monitor the balloon so that it does not get sucked into the flask due to backpressure. Thereafter, pour the entire reaction mixture into a distillation apparatus, and distil-off the methanol and any excess ammonia. When no more methanol is collected, stop the distillation, and then remove the left over remaining residue (after it has cooled to room temperature), and then place it into a clean beaker. Thereafter add in 60 grams of 35 to 38% hydrochloric acid, and then stir the entire mixture for about 30 minutes. Then, briefly extract the entire acidic mixture with three 25-milliliter portions of diethyl ether, and after the extraction process, the ether portions can be discarded or recycled (as they will contain impurities). Now, to the extracted acidic mixture, add in a sodium hydroxide solution prepared by adding and dissolving 60 grams of sodium hydroxide into 170 milliliters of water. Note: sodium hydroxide generates excessive heat when dissolved in water, so allow the solution to cool to room temperature before using. After the addition of the sodium hydroxide solution, stir the entire alkaline mixture for about 30 minutes at room temperature. Then extract this entire alkaline mixture with three 75-milliliter portions of diethyl ether, and after the extraction process, combine all ether extracts (if not already done so), and then dry this combined ether portion by adding to it, 15 grams of anhydrous magnesium sulfate. Then stir the whole mixture for about 10 minutes, and then filter-off the magnesium sulfate. Then place this filtered ether mixture into an ice bath, and chill to 0 Celsius. Then bubble into this ether mixture, 15 grams of dry hydrogen chloride gas (excess), and after the addition, stir the entire ether mixture at 0 Celsius for about 1 hour. Then filter-off the precipitated MDA hydrochloride product, and then vacuum dry or air-dry the crystals. These crystals can be recrystallized from a methylene chloride/petroleum ether mixture or from any suitable solvent if desired. **Note:** the pressure process discussed earlier, whereby a balloon is placed over a flask, can be substituted by using a conventional steel pipe with threads at both ends. To carryout the steel pipe technique, pour all necessary materials (as describe for the balloon technique), into a thick walled stainless steel pipe, and then seal both ends with the corresponding steel caps. The threads at each end should be wrapped with Teflon tape prior to screwing in the end caps. Then place the entire pipe, and submerge it into an oil bath and heat at the desired temperature for the desired amount of time. Note: this process can be dangerous and can lead to pressure explosions. Carryout the process in an area that can contain any such explosion, and maintain a safe distance away during the operation—just to be on the safe side.

Note: Other salts of the freebase MDA such as the sulfate, tartrate, citrate, and phosphate can be prepared by adding the corresponding acid to the ether mixture of the MDA freebase compound obtained at the end of the procedure. For sulfuric acid or tartaric acid, 1 mole of 98% sulfuric acid or d-tartaric acid should be added to 2 moles of the ether mixture of the MDA freebase. For citric acid or phosphoric acid, 1 mole of the acid should be added to 3 moles of the ether mixture of the MDA freebase. The ether mixture after treatment with the corresponding acid in each of these cases can then be filtered to recover the precipitated crystals of the desired product. All the salts of MDA are stimulants and are psychedelic in nature. The tartrate and citrate salts may be twice as potent as the hydrochloride or sulfate.

Procedure D: Preparation of MDA hydrochloride from piperonylacetone using aluminum amalgam

Materials:

1. 40 grams of sodium hydroxide	8. 68 milliliters of a 25% sodium hydroxide solution
2. 20 grams of aluminum foil	9. 24.7 grams of piperonylacetone (see Intermediate-0011. Piperonylacetone)
3. 100 milliliters of 95% ethyl alcohol or 100 milliliters of denatured alcohol	10. 100 milliliters of methanol
4. 500 milligrams of mercury-II-chloride	11. 280 milliliters of 10% hydrochloric acid
5. 366 milliliters of diethyl ether	12. 495 milliliters of methylene chloride
6. 22 grams of ammonium chloride	13. 15 grams of anhydrous magnesium sulfate
7. 247 milliliters of 99% isopropyl alcohol	14. 20 grams of hydrogen chloride gas

Summary: MDA hydrochloride can be prepared by reacting piperonylacetone with amalgated aluminum. The reaction is generally mild, and afterwards, the reaction mixture is filtered, evaporated to remove solvents and water, and then extracted into hydrochloric acid, from where it forms the water-soluble hydrochloride. The hydrochloride is then purified by extraction of the freebase oil into methylene chloride by addition of sodium hydroxide, which liberates this freebase oil. The resulting methylene chloride mixture is then evaporated to remove the methylene chloride, and the left over oil is then dissolved into ether, whereby it is finally precipitated as the purified MDA hydrochloride.

Hazards: Extinguish all flames before using diethyl ether, which is highly flammable, and can form explosive mixtures with air. Wear gloves when handling mercury chloride, and any mercury containing solutions, or mixtures, as they can be absorbed into the skin. Ethyl alcohol, methanol, and isopropyl alcohol are flammable, and methanol burns with a colorless flame, so use caution. Sodium hydroxide, hydrochloric acid, and dry hydrogen chloride gas are corrosive, and capable of causing skin burns. Avoid inhalation of hydrogen chloride gas.

Procedure:

Personnel notes for procedure D: MDA hydrochloride

Step 1: Amalgamation of aluminum

Into a suitable beaker, place 90 milliliters of distilled water, followed by 10 grams of sodium hydroxide. Thereafter stir the mixture to dissolve the sodium hydroxide. Note: much heat is generated when sodium hydroxide is dissolved in water, so allow the sodium hydroxide solution to cool to room temperature before using. Thereafter, add in 20 grams of aluminum foil pieces (cut into small squares), and allow the aluminum foil pieces to stand in the sodium hydroxide solution for about 20 minutes or until the evolution of hydrogen gas has drastically decreased. When the hydrogen gas evolution has almost ceased, filter-off the remaining pieces of aluminum, and then wash these collected pieces of aluminum with three 50-millilter portions of distilled water, followed by one portion of 50 milliliters of 95% ethyl alcohol (denatured alcohol can be used if desired). After the washing portion, allow the aluminum pieces to air-dry. When the pieces have air-dried, prepare a solution by adding and dissolving 500 milligrams of mercury-II-chloride (mercuric chloride) into 25 milliliters of water. Thereafter, add to the mercury chloride solution, the air-dried aluminum pieces, and allow the mixture to stand for about 15 minutes. After 15 minutes, filter-off the insoluble amalgated aluminum pieces, and then wash these filtered-off pieces with two 50-milliliter portions of distilled water, followed by one 50-milliliter portion of 95% ethyl alcohol (denatured alcohol will work if desired), and then wash with one portion of 10 milliliters of diethyl ether. After the washings, store the amalgated aluminum pieces submerged in a small amount of diethyl ether until use.

Step 2: Preparation of MDA hydrochloride

Into a suitable flask or beaker, add in the amalgated aluminum prepared in step 1, followed by 22 grams of ammonium chloride dissolved in water (prepared by adding and dissolving the ammonium chloride into 30 milliliters of water), followed by 84 milliliters of 99% isopropyl alcohol, followed by 68 milliliters of a 25% sodium hydroxide solution, followed by 24.7 grams of piperonylacetone, and then followed by 163 milliliters of 99% isopropyl alcohol. Thereafter, moderately stir the reaction mixture for about 60 minutes. Note: during the reaction, keep the reaction mixtures temperature below 58 Celsius—a ice bath

or cold water bath may or may not be needed, but most likely will be needed, so place the flask or beaker into a ice bath or ice water bath prior to adding the ingredients. After stirring the reaction mixture for 60 minutes, filter the reaction mixture to remove insoluble materials. Note: instead of filtering using the normal methods, pour a layer of celite (diatomaceous silicate powder) over the filter paper before filtering the reaction mixture after the initial 60-minute period. After filtering, pass two 50-milliliter portions of methanol through the filter (containing the celite), and then combine these two methanol portions to the filtered reaction mixture, and then place the entire reaction mixture into a distillation apparatus, and distill at 100 Celsius to remove the methanol, isopropyl alcohol, and water. When no more methanol, isopropyl alcohol, or water passes over or is collected, stop the distillation process, and allow the left over remaining oily residue to cool to room temperature before collecting it. Thereafter, dissolve the recovered oily residue into 56 milliliters of diethyl ether, and then extract this ether mixture with two 140-millilter portions of 10% hydrochloric acid. Note: after the extraction process, the hydrochloric acid mixture will be the lower layer each time. After the extraction process, briefly extract this hydrochloric acid mixture with three 25-milliliter portions of methylene chloride (to remove impurities), and then discard or recycle the methylene chloride portions. Note: after each extraction, the methylene chloride portion will be the lower layer each time. After the extraction process, basify the hydrochloric acid mixture by adding to it, a sodium hydroxide solution prepared by adding and dissolving 30 grams of sodium hydroxide into 150 milliliters of water. Note: sodium hydroxide generates much heat when dissolved in water, so allow the solution to cool to room temperature before using. After adding the sodium hydroxide solution, moderately stir the alkaline mixture for about 30 minutes at room temperature. Finally, extract this alkaline mixture with three 140-milliliter portions of methylene chloride, and after the extraction process, combine all methylene chloride portions (if not already done so), and then dry this combined methylene chloride portion by adding to it, 15 grams of anhydrous magnesium sulfate. Then stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Then place this methylene chloride mixture into a distillation apparatus, and remove it at 40 Celsius. When no more methylene chloride passes over, recover the left over remaining oily residue (after it has cooled), and then dissolve it into 300 milliliters of diethyl ether. Thereafter, place this ether mixture into an ice bath, and chill to 0 Celsius. Then bubble into this chilled ether mixture, 20 grams (excess) of hydrogen chloride gas, and after the addition of the hydrogen chloride, stir the entire mixture for about 30 minutes at 0 Celsius. Then filter-off the precipitated MDA hydrochloride product, and then vacuum dry or air-dry the crystals.

Note: Other salts of the freebase MDA such as the sulfate, tartrate, citrate, and phosphate can be prepared by adding the corresponding acid to the ether mixture of the MDA freebase compound obtained at the end of step 2. For sulfuric acid or tartaric acid, 1 mole of 98% sulfuric acid or d-tartaric acid should be added to 2 moles of the ether mixture of the MDA freebase. For citric acid or phosphoric acid, 1 mole of the acid should be added to 3 moles of the ether mixture of the MDA freebase. The ether mixture after treatment with the corresponding acid in each of these cases can then be filtered to recover the precipitated crystals of the desired product. All the salts of MDA are stimulants and are psychedelic in nature. The tartrate and citrate salts may be twice as potent as the hydrochloride or sulfate.

Procedure E: Preparation of MDA hydrochloride

Materials:

1. 210 grams of 28 to 30% ammonia solution	5. 600 milliliters of diethyl ether
2. 180 grams of 80% formic acid solution	6. 100 grams of sodium hydroxide
3. 120 grams of piperonylacetone (see Intermediate-0011. Piperonylacetone)	7. 25 grams of anhydrous magnesium sulfate
4. 240 milliliters of 35 to 38% hydrochloric acid	8. 30 grams of dry hydrogen chloride gas

Summary: In this process, MDA hydrochloride is formed by the reaction of piperonylacetone with concentrated ammonia solution in the presence of concentrated formic acid. The reaction is carried out at a temperature of about 160 Celsius. After the initial reaction period, the reaction mixture is hydrolyzed with concentrated hydrochloric acid, and the reaction mixture is refluxed at 100 Celsius to properly carryout the hydrolysis. After the hydrolysis, the reaction mixture is diluted with water, and then briefly extracted with ether to dissolve impurities. The reaction mixture, after being extracted, is then basified by the addition of sodium hydroxide. After which, the alkaline reaction mixture is then extracted to dissolve the freebase oil. The combined ether portions are then treated with hydrogen chloride gas to precipitate the MDA hydrochloride.

Hazards: Extinguish all flames before using diethyl ether, which is highly flammable and can form explosive mixtures with air. Wear gloves and use maximum ventilation when handling concentrated ammonia solutions, as they are highly irritating to the eyes, nose, and throat. Avoid prolonged exposure to ammonia vapors. Concentrated formic acid, sodium hydroxide, and hydrochloric acid are capable of forming skin burns and irritation, so wear gloves when handling. Use proper ventilation when handling concentrated hydrochloric acid, which is a highly fuming substance.

Procedure:

Personnel notes for procedure E: MDA hydrochloride

Into a standard reflux apparatus with a 3-neck flask (the 3-neck flask should be equipped with thermometer, motorized stirrer, and addition funnel), place 210 grams of 28 to 30% ammonia solution, followed by 180 grams of 80% formic acid solution. Thereafter, heat the contents in the 3-neck flask to about 160 Celsius, and when the temperature reaches 160 Celsius, add in all at once, 120 grams of piperonylacetone. Then reflux the entire reaction mixture at 160 Celsius for about 14 hours. During the reflux period, moderately stir the reaction mixture. After 14 hours, reduce the heat to about 100 Celsius, and immediately after the temperature reaches 100 Celsius, place into the addition funnel, 240 milliliters of 35 to 38% hydrochloric acid (muriatic acid will work—31% hydrochloric acid), and then add this hydrochloric acid to the reaction mixture over a period of about 10 minutes. During the addition of the acid, continue to heat the reaction mixture at about 100 Celsius with moderate stirring. After 10 minutes, continue to reflux the reaction mixture at 100 Celsius under moderate stirring for 16 hours. After 16 hours, remove the heat source, and allow the reaction mixture to cool to room temperature. Thereafter, pour the entire reaction mixture into a suitable sized beaker, and then add in 400 milliliters of water. Thereafter, stir the entire reaction mixture for about 15 minutes. Then briefly extract the entire diluted reaction mixture with three 50-millilter portions of diethyl ether (to remove impurities), and after the extraction process, discard or recycle the ether portions. Note: after the extraction process, the ether will be the upper layer each time. Now, to the extracted reaction mixture, add in a sodium hydroxide solution prepared by adding and dissolving 100 grams of sodium hydroxide into 300 milliliters of water. Note: sodium hydroxide generates much heat when dissolved in water, so allow the sodium hydroxide solution to cool to room temperature before using it. After the addition of the sodium hydroxide solution, stir the entire alkaline mixture for about 30 minutes. Finally, extract this alkaline mixture with three 150-milliliter portions of diethyl ether, and then after the extraction process, combine all ether portions, if not already done so, and then wash this combined ether portion with three 100-milliliter portions of cold water. Note: after the extraction, and washings, the ether will be the upper layer each time. After the washing process, dry the washed ether portion by adding to it, 25 grams of anhydrous magnesium sulfate, and then stir the entire ether mixture for about 10 minutes—then filter-off the magnesium sulfate. Finally, place the filtered ether portion into an ice bath, and chill to 0 Celsius. Thereafter, bubble into the ether mixture, 30 grams of dry hydrogen chloride gas (an excess). After the addition of the hydrogen chloride gas, continue to stir the ether mixture for 1 hour, and then filter-off the precipitated MDA hydrochloride product, and then vacuum dry or air-dry the crystals.

Note: Other salts of the freebase MDA such as the sulfate, tartrate, citrate, and phosphate can be prepared by adding the corresponding acid to the ether mixture of the MDA freebase compound obtained at the end of the above process. For sulfuric acid or tartaric acid, 1 mole of 98% sulfuric acid or d-tartaric acid should be added to 2 moles of the ether mixture of the MDA freebase. For citric acid or phosphoric acid, 1 mole of the acid should be added to 3 moles of the ether mixture of the MDA freebase. The ether mixture after treatment with the corresponding acid in each of these cases can then be filtered to recover the precipitated crystals of the desired product. All the salts of MDA are stimulants and are psychedelic in nature. The tartrate and citrate salts may be twice as potent as the hydrochloride or sulfate.

Procedure F: Preparation of MDA hydrochloride from piperonal

Materials:

1. 7.5 grams of piperonal (see intermediate-0011, procedure B, of step 3, and intermeidate-0024 for its preparation)	9. 150 milliliters of a 5% sulfuric acid solution
2. 140 milliliters of glacial acetic acid	10. 195 milliliters of methylene chloride
3. 7.5 milliliters of nitroethane	11. 100 grams of a 15% sodium hydroxide solution
4. 5 grams of cyclohexylamine	12. 15 grams of sodium hydroxide
5. 8 grams of lithium aluminum hydride	13. 15 grams of anhydrous sodium sulfate
6. 250 milliliters of tetrahydrofuran (THF)	14. 20 grams of 35 to 38% hydrochloric acid solution
7. 30 milliliters of 99% isopropyl alcohol	15. 100 milliliters of diethyl ether
8. 6 milliliters of a 15% sodium hydroxide solution	

Summary: MDA hydrochloride can be prepared directly from piperonal by first, condensation of the piperonal with nitroethane in the presence of glacial acetic acid. The reaction is identical to all the other nitro condensations in this book. After the reaction, the mixture is treated with water, and then allowed to sit overnight to precipitate the nitro intermediate. Once the nitro intermediate has been collected, it is converted into the desired product by reaction with lithium aluminum hydride in the usual manner. The reaction is rather general, and afterwards, it is treated with alcohol and sodium hydroxide to destroy

impurities, and the filtered reaction mixture is then thereby treated with acid, and the resulting acidified mixture is then quickly extracted with methylene chloride to remove impurities. The resulting acidified mixture is then basified, and then extracted to recover the freebase. The methylene chloride extract is then stripped of solvent, and the final left over residue is then dissolved in alcohol, and then treated with acid. The desired MDA hydrochloride is then precipitated by the addition of ether.

Hazards: Be sure to extinguish all flames before using diethyl ether, tetrahydrofuran, and nitroethane, both of which are highly flammable, and capable of producing explosive mixtures with air. Wear gloves when handling sodium hydroxide, sulfuric acid, hydrochloric acid, and glacial acetic acid, all of which are capable of producing skin burns and irritation. Lithium aluminum hydride is very reactive, and dangerous in contact with water—use caution.

Procedure:

Personnel notes for procedure F: MDA hydrochloride

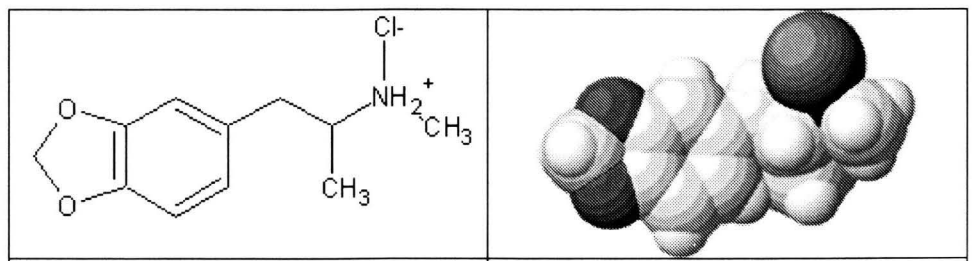
Into a standard reflux apparatus, equipped with stirring means, and thermometer, place 7.5 grams of piperonal (see intermediate-0011, procedure B, of step 3, and intermeidate-0024 for its preparation), followed by 40 milliliters of glacial acetic acid. Thereafter, stir the entire mixture for about 5 minutes, and then add in 7.5 milliliters of nitroethane, followed immediately by 5 grams of cyclohexylamine as catalyst. Then reflux the entire mixture at 100 Celsius for about 3 hours with constant stirring. After 3 hours, remove the heat source and allow the reaction mixture to cool to room temperature. Thereafter, pour this entire reaction mixture into a suitable sized beaker, and then add in 50 milliliters of ice-cold water. Thereafter, place this diluted reaction mixture into an ice bath, and allow it to stand overnight at 0 Celsius. Note: the reaction mixture can be stored in a freezer if desired. The next day, filter-off the precipitated crystals, and then vacuum dry or air-dry the crystals. The resulting dried crystals can then be recrystallized from 100 milliliters of glacial acetic acid. After the recrystallization process, vacuum dry or air-dry the crystals of the nitro intermediate.

Now, into a suitable 3-neck flask equipped with thermometer, reflux condenser (attach a calcium chloride drying tube to the top the reflux condenser to keep moisture out), addition funnel, and stirring means, place 8 grams of lithium aluminum hydride, followed by 100 milliliters of tetrahydrofuran (THF). Thereafter, stir the entire mixture for about 10 minutes to dissolve all solids. Then prepare a solution by adding and dissolving 7.2 grams of the nitro intermediate (prepared in the above paragraph), into 100 milliliters of tetrahydrofuran, and then place this solution into the addition funnel. Then gradually add this solution (from the addition funnel), drop-wise, over a period sufficient to keep the reaction mixture below 10 Celsius at all times. After the addition, reflux the entire reaction mixture at 66 Celsius for about 14 hours. After refluxing for about 14 hours, remove the heat source, and then allow the reaction mixture to cool to room temperature. Then pour the entire reaction mixture into a clean beaker, and then add in 6 milliliters of 99% isopropyl alcohol, followed by 6 milliliters of a 15% sodium hydroxide solution. After both additions, rapidly stir the reaction mixture. Thereafter, add in 20 milliliters of cold water, and then briefly stir the entire mixture for about 10 minutes. Then quickly filter-off the insoluble solids, and then quickly wash these filtered-off solids with two 25-milliliter portions of tetrahydrofuran, and after the washings, combine both tetrahydrofuran portions with the filtered reaction mixture. Then place this total combined reaction mixture into a distillation apparatus, and distill-off the tetrahydrofuran at 66 Celsius. When no more tetrahydrofuran passes over or is collected, stop the distillation process, and then recover the left over remaining residue (after it has cooled), and then dissolve this left over residue into 150 milliliters of a 5% sulfuric acid solution. Then rapidly stir the entire acidic mixture for about 30 minutes, and then extract this acidic mixture with three 25-millilter portions of methylene chloride (to remove impurities), and after the extraction process, the methylene chloride portions can be recycled or discarded if desired. Note: after each extraction process, the methylene chloride will be the upper layer each time. After the extraction process, basify the lower aqueous acidic layer by adding to it, 100 grams of a 15% sodium hydroxide solution prepared by adding and dissolving 15 grams of sodium hydroxide into 85 milliliters of cold water. Note: sodium hydroxide generates excessive heat when dissolved in water, so allow the solution to cool before using. After the addition of the sodium hydroxide solution, rapidly stir the mixture for about 30 minutes, and then extract the entire mixture with three 40-milliliter portions of methylene chloride. After the extraction process, combine all methylene chloride portions (if not already done so), and then dry this combined methylene chloride portion by adding to it, 15 grams of anhydrous sodium sulfate. Then stir the entire mixture for about 10 minutes, and then filter-off the sodium sulfate. Then place this filtered methylene chloride portion into a distillation apparatus, and distill-off the methylene chloride at 40 Celsius. When no more methylene chloride passes over or is collected, stop the distillation process, and remove the left over remaining oily residue (after it has cooled). Then place this left over remaining oily residue into a suitable beaker, and then add in 24 milliliters of 99% isopropyl alcohol. Then rapidly stir the entire alcohol mixture for about 30 minutes, and then quickly filter-off any insoluble materials. Thereafter, add to this filtered alcohol mixture, 20 grams of 35 to 38% hydrochloric acid (muriatic acid of

31% will work), and then stir the entire acidic alcohol mixture for about 10 minutes. Finally, add in, 50 milliliters of diethyl ether, and then rapidly stir the mixture for about 10 minutes. Thereafter, allow the entire mixture to stand overnight. The following day, filter-off the precipitated crystals, wash with two 25-milliliter portions of diethyl ether, and then vacuum dry or air-dry the crystals. The result will be about 4 grams of the desired product of MDA hydrochloride.

Note: Other salts of the freebase MDA such as the sulfate, tartrate, citrate, and phosphate can be prepared in the usual manner by adding the corresponding acid to the alcohol mixture of the MDA freebase compound obtained at the end of the above process. For sulfuric acid or tartaric acid, 1 mole of 98% sulfuric acid or d-tartaric acid should be added to 2 moles of the alcohol mixture of the MDA freebase (on a dry basis of the dry MDA freebase, not the total weight of the alcohol mixture). For citric acid or phosphoric acid, 1 mole of the acid should be added to 3 moles of the alcohol mixture of the MDA freebase. The alcohol mixture after treatment with the corresponding acid in each of these cases can then be treated with 2 volumes of ether per 1 volume of the alcohol mixture. Thereafter, the entire mixture is left to stand overnight, and the following day, filtered to recover the desired precipitated product. As usual, all the salts of MDA are stimulants and are psychedelic in nature. The tartrate and citrate salts may be twice as potent as the hydrochloride or sulfate.

0013. MDMA. Ecstasy. 3,4-Methylenedioxymethamphetamine hydrochloride. *1-(1,3-benzodioxol-5-yl)propan-2-amine hydrochloride*



Structure	3D model

Ecstasy is an interesting compound with hallucinogenic and psychedelic effects similar to, but with much less frequency then LSD. Ecstasy has been explained by some to produce an extra ordinary array of effects ranging from feelings of well being, stimulation, intoxification effects, mild and/or fluctuating hallucinogens (the degrees of which may differ), and a number of other physical effects. The exact effects and feelings of the drug experienced by consumers may vary along with other factors including dosage, alcohol intake, and/or other drug interactions. Ecstasy's use by persons is increasing, and it's a popular "party" drug. Some have explained feeling extra ordinary psychedelic effects with the drug in combination with alcohol. Overall, ecstasy is a simple amphetamine derivative, but with less "upper" effects encountered by the amphetamines. In essence, the drug produces stimulation, an increase in motivation, energy and awareness, and intoxification, all while in combination with unique physical enhancements, many of which are difficult to explain.

Note: This substance is a controlled substance (hallucinogen/stimulant) as listed in the US code of Federal regulations.

Toxicity: Low	**Rate of onset (average):** Rapid
Stimulation dosage (ingestion): 80 to 150 milligrams	**Duration of effects (average):** 4 to 6 hours (depending on the person)
Stimulation dosage (inhalation): 45 to 95 milligrams	**Habit forming potential:** Moderate
Stimulation dosage (injection): 35 milligrams +	**Estimated value U.S. (based on procedure):** $15 per gram

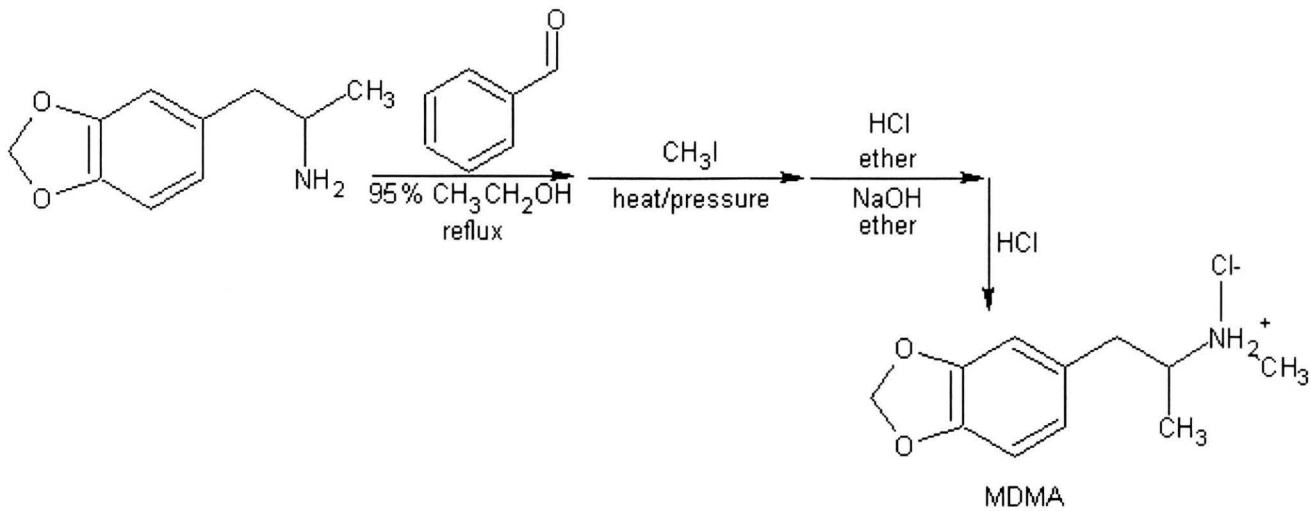
Procedure A: Preparation of MDMA

Materials:

1. 50 grams of MDA hydrochloride (see 0012. MDA hydrochloride)	6. 250 milliliters of 95% ethyl alcohol
2. 65 grams of sodium hydroxide	7. 28.4 grams of methyl iodide
3. 750 milliliters of diethyl ether	8. 20 milliliters of methanol
4. 30 grams of anhydrous magnesium sulfate	9. 40 grams of 35 to 38% hydrochloric acid
5. 21.2 grams of benzaldehyde	10. 12 grams of dry hydrogen chloride gas

Summary: MDMA is formed by the reaction of MDA with methyl iodide under heat and pressure. The reaction is rather general, and afterwards, the desired MDMA is recovered from the reaction mixture by acidifying the reaction mixture, followed by extraction with ether (to remove impurities), and then treatment of the extracted reaction mixture with sodium

hydroxide to liberate the freebase. The freebase is then collected by extraction into ether, and the resulting ether mixture is then treated with hydrogen chloride to precipitate the MDMA.



MDMA

Hazards: Extinguish all flames before using diethyl ether, which is highly flammable, and can form explosive mixtures with air. Use care when handling hydrogen chloride gas, which is very irritating to the nose and throat. 95% Ethyl alcohol, and methanol are both flammable, so keep away from fire. Note: methanol burns with a colorless flame, and burning methanol can be hard to see, especially outdoors.

Procedure:

Personnel notes for procedure A: MDMA

Place 50 grams of MDA hydrochloride into a beaker, and then add in a sodium hydroxide solution prepared by adding and dissolving 15 grams of sodium hydroxide into 50 milliliters of water. Note: sodium hydroxide generates much heat when dissolved in water, so allow the solution to cool to room temperature before using. After the addition of the sodium hydroxide, stir the entire mixture for 1 hour at ambient temperature (room temperature). Thereafter, extract the entire mixture with three-100 milliliter portions of diethyl ether, and after the extraction process, combine all ether portions (if not already done so), and then dry this combined ether portion by adding to it, 15 grams of anhydrous magnesium sulfate. Then stir the entire ether mixture for 10 minutes, and then filter-off the magnesium sulfate. Then place the filtered ether mixture into a distillation apparatus or rotary evaporator, and remove the ether. When no more ether distills over, recover the left over remaining oil residue (after it has cooled to room temperature), and then place 35.8 grams of this oil residue into a clean suitable reflux apparatus. Shortly thereafter, add in 21.2 grams of benzaldehyde, followed by 250 milliliters of 95% ethyl alcohol. Note: more ethyl alcohol may be needed to dissolve all the products—make sure all products are dissolved into the ethyl alcohol before proceeding. Thereafter, reflux the entire mixture for about 15 minutes at 78 Celsius. After the refluxing period, quickly remove the reflux condenser and replace it with a conventional condenser (fitted with a receiver flask), and then distill-off the ethyl alcohol. When no more ethyl alcohol passes over, stop the distillation process, and recover the left over remaining residue (after it has cooled). Now, place this left over residue into a suitable sized single neck flask, and then add in 28.4 grams of methyl iodide. Thereafter, place a suitable sized balloon over the flask, and secure the balloon to the flask using a metal ring clamp. Then heat the contents in the flask to about 100 Celsius for 5 hours. Note: the balloon will inflate and deflate sporadically during the heating process. The balloon is deigned to keep the contents of the flask under pressure to properly carryout the reaction. If during the heating process, the balloon pops or explodes, quickly replace with another one, and continue the operation for the necessary amount of remaining time. ***Note:*** *the pressure process just described, whereby a balloon is placed over a flask, can be substituted by using a conventional steel pipe with threads at both ends. To carryout the steel pipe technique, pour all necessary materials (as described for the balloon technique), into a thick walled stainless steel pipe, and then seal both ends with the corresponding steel caps. The threads at each end should be wrapped with Teflon tape prior to screwing in the end caps. Then place the entire pipe, and submerge it into a water bath and heat at the desired temperature for the desired time. Note: this process can be dangerous and can lead to pressure explosions. Carryout the process in an area that can contain any such explosion, and maintain a safe distance away during the operation—just to be on*

the safe side. After the heating process, remove the heat source, and allow the reaction mixture to cool to room temperature. Note: monitor the balloon so it does not get sucked into the flask due to backpressure. Thereafter, remove the contents of the flask, and place them into a clean beaker. Then add in 20 milliliters of methanol, followed by 5 milliliters of water, and then stir the entire mixture for about 30 minutes. Then place this entire mixture into a clean reflux apparatus, and then reflux the entire mixture at 68 Celsius for 30 minutes. After refluxing for 30 minutes, quickly add in 100 milliliters of water, and then continue the reflux at 68 Celsius for 30 additional minutes. Thereafter, remove the heat source, and allow the refluxed mixture to cool to room temperature. Then pour the entire refluxed mixture into a clean suitable sized beaker, and then add in 40 grams of 35 to 38% hydrochloric acid, and then stir the entire mixture for about 30 minutes. Afterwards, quickly extract the entire mixture (to remove impurities) with three 50-milliliter portions of diethyl ether, and after the extraction process, the ether portions can be discard or recycled if desired. Now, to the extracted acidic mixture, add in a sodium hydroxide solution prepared by adding and dissolving 50 grams of sodium hydroxide into 190 milliliters of water. Note: sodium hydroxide generates much heat when dissolved in water, so allow the alkaline solution to cool to room temperature before using. Then stir the entire mixture for 30 minutes, and thereafter, extract the entire alkaline mixture with three 100-milliliter portions of diethyl ether. After the extraction process, combine all ether portions (if not already done so), and then wash this combined ether portion with three 100-milliliter portions of cold water. Note: during the extraction and washing, the ether will be the upper layer each time. After the washing period, dry the washed ether portion by adding to it, 15 grams of anhydrous magnesium sulfate, and then stir the whole mixture for about 10 minutes—thereafter, filter-off the magnesium sulfate. Finally, place the filtered ether mixture into an ice bath, and chill to 0 Celsius. Then bubble into the ether mixture, 12 grams of dry hydrogen chloride gas (an excess), and after the addition of the hydrogen chloride, filter-off the precipitated MDMA product, and then vacuum dry or air-dry the crystals.

Note: Other salts of ecstasy (besides the hydrochloride) such as the sulfate, tartrate, citrate, and phosphate can be prepared by adding the corresponding acid to the ether mixture of the extracted freebase compound obtained at the end of the above procedure. For sulfuric acid or tartaric acid, 1 mole of 98% sulfuric acid or d-tartaric acid should be added to 2 moles of the ether mixture of the extracted freebase. For citric acid or phosphoric acid, 1 mole of the acid should be added to 3 moles of the ether mixture of the extracted freebase. The ether mixture after treatment with the corresponding acid in each of these cases can then be filtered to recover the precipitated crystals of the desired product. All the salts of Ecstasy (other then the hydrochloride) are hallucinogens/stimulants and are psychedelic in nature. The tartrate and citrate salts may be twice as potent as the original hydrochloride (ecstasy).

Procedure B: Preparation of MDMA from piperonylacetone via amalgated aluminum reduction

Materials:

1. 40 grams of sodium hydroxide	8. 68 milliliters of a 25% sodium hydroxide solution
2. 20 grams of aluminum foil	9. 24.7 grams of piperonylacetone (see Intermeidate-0011. Piperonylacetone)
3. 100 milliliters of 95% ethyl alcohol or 100 milliliters of denatured alcohol	10. 100 milliliters of methanol
4. 500 milligrams of mercury-II-chloride	11. 280 milliliters of 10% hydrochloric acid
5. 366 milliliters of diethyl ether	12. 495 milliliters of methylene chloride
6. 28 grams of methylamine hydrochloride	13. 15 grams of anhydrous magnesium sulfate
7. 247 milliliters of 99% isopropyl alcohol	14. 20 grams of hydrogen chloride gas

Summary: MDMA can be prepared by reacting piperonylacetone with amalgated aluminum in the presence of methylamine hydrochloride. The reaction is identical to that in procedure D for the preparation of MDA hydrochloride, and afterwards the reaction mixture is filtered, evaporated to remove solvents and water, and then extracted into hydrochloric acid, from where it forms the water-soluble hydrochloride. The hydrochloride is then purified by extraction of the freebase oil into methylene chloride by addition of sodium hydroxide, which liberates this freebase oil. The resulting methylene chloride mixture is then evaporated to remove the methylene chloride, and the left over oil is then dissolved into ether, whereby it is finally precipitated as the purified MDMA.

Hazards: Extinguish all flames before using diethyl ether, which is highly flammable, and can form explosive mixtures with air. Wear gloves when handling mercury chloride, and any mercury containing solutions, or mixtures, as they can be absorbed into the skin. Ethyl alcohol, methanol, and isopropyl alcohol are flammable, and methanol burns with a colorless flame, so use caution. Sodium hydroxide, hydrochloric acid, and dry hydrogen chloride gas are corrosive, and capable of causing skin burns. Avoid inhalation of hydrogen chloride gas.

Procedure:

Personnel notes for procedure B: MDMA

Step 1: Amalgamation of aluminum

Into a suitable beaker, place 90 milliliters of distilled water, followed by 10 grams of sodium hydroxide. Thereafter stir the mixture to dissolve the sodium hydroxide. Note: much heat is generated when sodium hydroxide is dissolved in water, so allow the sodium hydroxide solution to cool to room temperature before using. Thereafter, add in 20 grams of aluminum foil pieces (cut into small squares), and allow the aluminum foil pieces to stand in the sodium hydroxide solution for about 20 minutes or until the evolution of hydrogen gas has drastically decreased. When the hydrogen gas evolution has almost ceased, filter-off the remaining pieces of aluminum, and then wash these collected pieces of aluminum with three 50-millilter portions of distilled water, followed by one portion of 50 milliliters of 95% ethyl alcohol (denatured alcohol can be used if desired). After the washing portion, allow the aluminum pieces to air-dry. When the pieces have air-dried, prepare a solution by adding and dissolving 500 milligrams of mercury-II-chloride (mercuric chloride) into 25 milliliters of water. Thereafter, add to the mercury chloride solution, the air-dried aluminum pieces, and allow the mixture to stand for about 15 minutes. After 15 minutes, filter-off the insoluble amalgated aluminum pieces, and then wash these filtered-off pieces with two 50-milliliter portions of distilled water, followed by one 50-milliliter portion of 95% ethyl alcohol (denatured alcohol will work if desired), and then wash with one portion of 10 milliliters of diethyl ether. After the washings, store the amalgated aluminum pieces submerged in a small amount of diethyl ether until use.

Step 2: Preparation of MDMA

Into a suitable flask or beaker, add in the amalgated aluminum prepared in step 1, followed by 28 grams of methylamine hydrochloride dissolved in water (prepared by adding and dissolving the methylamine hydrochloride into 30 milliliters of water), followed by 84 milliliters of 99% isopropyl alcohol, followed by 68 milliliters of a 25% sodium hydroxide solution, followed by 24.7 grams of piperonylacetone, and then followed by 163 milliliters of 99% isopropyl alcohol. Thereafter, moderately stir the reaction mixture for about 60 minutes. Note: during the reaction, keep the reaction mixtures temperature below 58 Celsius—a ice bath or cold water bath may or may not be needed, but most likely will be needed, so place the flask or beaker into a ice bath or ice water bath prior to adding the ingredients. After stirring the reaction mixture for 60 minutes, filter the reaction mixture to remove insoluble materials. Note: instead of filtering using the normal methods, pour a layer of celite (diatomaceous silicate powder) over the filter paper before filtering the reaction mixture after the initial 60-minute period. After filtering, pass two 50-milliliter portions of methanol through the filter (containing the celite), and then combine these two methanol portions to the filtered reaction mixture, and then place the entire reaction mixture into a distillation apparatus, and distill at 100 Celsius to remove the methanol, isopropyl alcohol, and water. When no more methanol, isopropyl alcohol, or water passes over or is collected, stop the distillation process, and allow the left over remaining oily residue to cool to room temperature before collecting it. Thereafter, dissolve the recovered oily residue into 56 milliliters of diethyl ether, and then extract this ether mixture with two 140-millilter portions of 10% hydrochloric acid. Note: after the extraction process, the hydrochloric acid mixture will be the lower layer each time. After the extraction process, briefly extract this hydrochloric acid mixture with three 25-milliliter portions of methylene chloride (to remove impurities), and then discard or recycle the methylene chloride portions. Note: after each extraction, the methylene chloride portion will be the lower layer each time. After the extraction process, basify the hydrochloric acid mixture by adding to it, a sodium hydroxide solution prepared by adding and dissolving 30 grams of sodium hydroxide into 150 milliliters of water. Note: sodium hydroxide generates much heat when dissolved in water, so allow the solution to cool to room temperature before using. After adding the sodium hydroxide solution, moderately stir the alkaline mixture for about 30 minutes at room temperature. Finally, extract this alkaline mixture with three 140-milliliter portions of methylene chloride, and after the extraction process, combine all methylene chloride portions (if not already done so), and then dry this combined methylene chloride portion by adding to it, 15 grams of anhydrous magnesium sulfate. Then stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Then place this methylene chloride mixture into a distillation apparatus, and remove it at 40 Celsius. When no more methylene chloride passes over, recover the left over remaining oily residue (after it has cooled), and then dissolve it into 300 milliliters of diethyl ether. Thereafter, place this ether mixture into an ice bath, and chill to 0 Celsius. Then bubble into this chilled ether mixture, 20 grams (excess) of hydrogen chloride gas, and after the addition of the hydrogen chloride, stir the entire mixture for about 30 minutes at 0 Celsius. Then filter-off the precipitated MDMA product, and then vacuum dry or air-dry the crystals.

Note: Other salts of ecstasy (besides the hydrochloride) such as the sulfate, tartrate, citrate, and phosphate can be prepared by adding the corresponding acid to the ether mixture of the extracted freebase compound obtained at the end of step 2. For sulfuric acid or tartaric acid, 1 mole of 98% sulfuric acid or d-tartaric acid should be added to 2 moles of the ether mixture of

the extracted freebase. For citric acid or phosphoric acid, 1 mole of the acid should be added to 3 moles of the ether mixture of the extracted freebase. The ether mixture after treatment with the corresponding acid in each of these cases can then be filtered to recover the precipitated crystals of the desired product. All the salts of ecstasy (other then the hydrochloride) are hallucinogens/stimulants and are psychedelic in nature. The tartrate and citrate salts may be twice as potent as the original hydrochloride (ecstasy).

Procedure C: Preparation of MDMA

Materials:

1. 69 grams of sodium hydroxide	5. 60 milliliters of 35 to 38% hydrochloric acid
2. 58 grams of methylamine hydrochloride	6. 375 milliliters of diethyl ether
3. 50 grams of 80% formic acid solution	7. 15 grams of anhydrous magnesium sulfate
4. 30 grams of piperonylacetone (see Intermeidate-0011. Piperonylacetone)	8. 20 grams of dry hydrogen chloride gas

Summary: In this process, MDMA is formed in an identical manner as for procedure E for the preparation of MDA hydrochloride; however, the ammonia is replaced with methylamine. The MDMA is formed by the reaction of piperonylacetone with a 40% methylamine solution in the presence of concentrated formic acid. The reaction mixture is heated at 160 Celsius for proper reaction temperature. After the initial reaction period, the reaction mixture is hydrolyzed with concentrated hydrochloric acid, and the reaction mixture is refluxed at 100 Celsius to properly carryout the hydrolysis. After the hydrolysis, the reaction mixture is diluted with water, and then briefly extracted with ether to dissolve impurities. The reaction mixture, after being extracted, is then basified by the addition of sodium hydroxide. After which, the alkaline reaction mixture is then extracted to dissolve the freebase oil. The combined ether portions are then treated with hydrogen chloride gas to precipitate the MDMA.

Hazards: Extinguish all flames before using diethyl ether, which is highly flammable and can form explosive mixtures with air. Concentrated formic acid, sodium hydroxide, and hydrochloric acid are capable of forming skin burns and irritation, so wear gloves when handling. Use proper ventilation when handling concentrated hydrochloric acid, which is a highly fuming substance.

Procedure:

Personnel notes for procedure C: MDMA

Into a suitable beaker, place a sodium hydroxide solution prepared by adding and dissolving 34 grams of sodium hydroxide into 40 milliliters of water. Note: sodium hydroxide generates much heat when dissolved in water, so allow the alkaline solution to cool to room temperature before using. Thereafter, slowly add in, 58 grams of methylamine hydrochloride. During the addition, keep the temperature of the sodium hydroxide solution below 30 Celsius. After the addition of the methylamine hydrochloride, stir the mixture for about 30 minutes at room temperature. Thereafter, filter the mixture to remove any insoluble solids. Then place this filtered mixture (which will contain a 40% solution of methylamine in water) into a standard reflux apparatus with a 3-neck flask (the 3-neck flask should be equipped with thermometer, motorized stirrer, and addition funnel), followed by 50 grams of 80% formic acid solution. Thereafter, heat the contents in the 3-neck flask to about 160 Celsius, and when the temperature reaches 160 Celsius, add in all at once, 30 grams of piperonylacetone. Then reflux the entire reaction mixture at 160 Celsius for about 3.5 hours. During the reflux period, moderately stir the reaction mixture. After 3.5 hours, reduce the heat to about 100 Celsius, and immediately after the temperature reaches 100 Celsius, place into the addition funnel, 60 milliliters of 35 to 38% hydrochloric acid (muriatic acid will work—31% hydrochloric acid), and then add this hydrochloric acid to the reaction mixture over a period of about 10 minutes. During the addition of the acid, continue to heat the reaction mixture at about 100 Celsius with moderate stirring. After 10 minutes, continue to reflux the reaction mixture at 100 Celsius under moderate stirring for 4 hours. After 4 hours, remove the heat source, and allow the reaction mixture to cool to room temperature. Thereafter, pour the entire reaction mixture into a suitable sized beaker, and then add in 100 milliliters of water. Thereafter, stir the entire reaction mixture for about 15 minutes. Then briefly extract the entire diluted reaction mixture with three 25-millilter portions of diethyl ether (to remove impurities), and after the extraction process, discard or recycle the ether portions. Note: after the extraction process, the ether will be the upper layer each time. Now, to the extracted reaction mixture, add in a sodium hydroxide solution prepared by adding and dissolving 35 grams of sodium hydroxide into 100 milliliters of

water. Note: sodium hydroxide generates much heat when dissolved in water, so allow the sodium hydroxide solution to cool to room temperature before using it. After the addition of the sodium hydroxide solution, stir the entire alkaline mixture for about 30 minutes. Finally, extract this alkaline mixture with three 100-milliliter portions of diethyl ether, and then after the extraction process, combine all ether portions, if not already done so, and then wash this combined ether portion with three 50-milliliter portions of cold water. Note: after the extraction, and washings, the ether will be the upper layer each time. After the washing process, dry the washed ether portion by adding to it, 15 grams of anhydrous magnesium sulfate, and then stir the entire ether mixture for about 10 minutes—then filter-off the magnesium sulfate. Finally, place the filtered ether portion into an ice bath, and chill to 0 Celsius. Thereafter, bubble into the ether mixture, 20 grams of dry hydrogen chloride gas (an excess). After the addition of the hydrogen chloride gas, continue to stir the ether mixture for 1 hour, and then filter-off the precipitated MDMA product, and then vacuum dry or air-dry the crystals.

Note: Other salts of ecstasy (besides the hydrochloride) such as the sulfate, tartrate, citrate, and phosphate can be prepared by adding the corresponding acid to the ether mixture of the extracted freebase compound obtained at the end of the above process. For sulfuric acid or tartaric acid, 1 mole of 98% sulfuric acid or d-tartaric acid should be added to 2 moles of the ether mixture of the extracted freebase. For citric acid or phosphoric acid, 1 mole of the acid should be added to 3 moles of the ether mixture of the extracted freebase. The ether mixture after treatment with the corresponding acid in each of these cases can then be filtered to recover the precipitated crystals of the desired product. All the salts of ecstasy (other then the hydrochloride) are hallucinogens/stimulants and are psychedelic in nature. The tartrate and citrate salts may be twice as potent as the original hydrochloride (ecstasy).

Procedure D: Preparation of MDMA directly from bromosafrole

Materials:

1. 24.5 grams of bromosafrole (see 0012. MDA hydrochloride, procedure A, step 1)	5. 350 milliliters of diethyl ether
2. 77.5 grams of a 40% methylamine solution	6. 55 grams of sodium hydroxide
3. 50 milliliters of methanol	7. 15 grams of anhydrous magnesium sulfate
4. 100 milliliters of 35 to 38% hydrochloric acid	8. 25 grams of dry hydrogen chloride gas

Summary: MDMA is prepared in a similar manner as for the preparation of MDA hydrochloride using a pressure apparatus. The reaction is quite simple and involves the amination of bromosafrole with methylamine under heat and pressure. After the reaction, the contents of the reaction mixture are distilled to remove solvent, and the resulting solvent free mixture is then acidified, briefly extracted with ether (to remove impurities), and the resulting extracted mixture is then basified with sodium hydroxide to liberate the freebase oil. The freebase oil is then dissolved into ether by extraction, and the extracted ether mixture is then treated with hydrogen chloride gas to precipitate the MDMA.

Hazards: Extinguish all flames before using diethyl ether, which is highly flammable, and can form explosive mixtures with air. Use proper ventilation when handling methylamine, which is a pungent irritating gas. Wear gloves when handling concentrated hydrochloric acid, and sodium hydroxide, both of which are capable of producing skin irritation.

Procedure:

Personnel notes for procedure D: MDMA

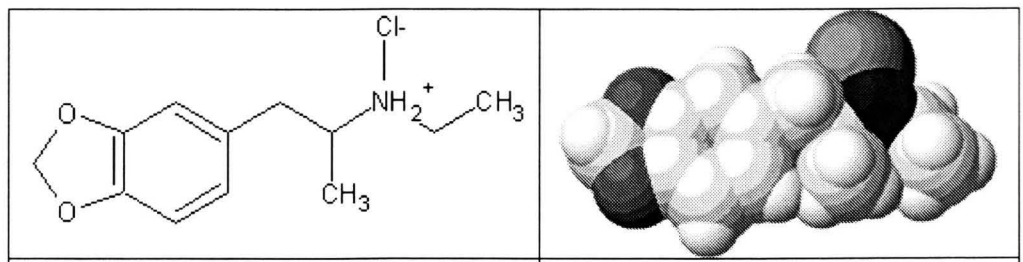
Into a suitable beaker, place 24.5 grams of bromosafrole, followed by 77.5 grams of a 40% methylamine solution. Thereafter, quickly add in 50 milliliters of methanol and then stir the entire mixture to ensure a uniform mixture. If after the addition of the methanol, two layers exist in the mixture, add a little more methanol until all layers disappear (merge). Thereafter, place the mixture into a suitable sized single neck flask, and then place a suitable sized balloon over the flask, and secure the balloon to the flask using a metal ring clamp. Then heat the contents in the flask to about 130 Celsius for 3 hours. Note: the balloon will inflate and deflate sporadically during the heating process. The balloon is deigned to keep the contents of the flask under pressure to properly carryout the reaction. If during the heating process, the balloon pops or explodes, quickly replace with another one, and continue the operation for the necessary amount of remaining time. ***Note:*** *the pressure process just described, whereby a balloon is placed over a flask, can be substituted by using a conventional steel pipe with threads at both ends. To carryout the steel pipe technique, pour all necessary materials (as described for the balloon technique), into a thick walled*

stainless steel pipe, and then seal both ends with the corresponding steel caps. The threads at each end should be wrapped with Teflon tape prior to screwing in the end caps. Then place the entire pipe, and submerge it into an oil bath and heat at the desired temperature for the desired time. Note: this process can be dangerous and can lead to pressure explosions. Carryout the process in an area that can contain any such explosion, and maintain a safe distance away during the operation—just to be on the safe side. After the heating process, remove the heat source, and allow the reaction mixture to cool to room temperature. Then pour the entire reaction mixture into a distillation apparatus, and distill-off the methanol at 68 Celsius. When no more methanol distills over, stop the distillation process, and recover the left over remaining residue (after it has cooled to room temperature). Thereafter, place the left over remaining residue into a beaker, and then add in 100 milliliters of 35 to 38% hydrochloric acid (muriatic acid will work—31%), and then stir the entire acidic mixture for about 30 minutes. Then briefly extract the acidic mixture with two 25-milliliter portions of diethyl ether (to remove impurities), and after the extraction process, discard or recycle the ether portions. Thereafter, add to the extracted acidic mixture, a sodium hydroxide solution prepared by adding and dissolving 55 grams (excess) of sodium hydroxide into 150 milliliters of water. Note: sodium hydroxide generates much heat when dissolved in water, so allow the alkaline mixture to cool to room temperature before using. After the addition of the sodium hydroxide solution, stir the entire alkaline mixture for about 30 minutes. Finally, extract this alkaline mixture with three 100-milliliter portions of diethyl ether, and after the extraction process, combine all ether extracts (if not already done so), and then dry the combined ether portion by adding to it, 15 grams of anhydrous magnesium sulfate, and then stir the entire mixture for about 10 minutes. Thereafter, filter-off the magnesium sulfate, and then place the filtered ether mixture into an ice bath, and chill to about 0 Celsius. Then bubble into the mixture 25 grams of dry hydrogen chloride gas (excess), and after the addition, stir the entire mixture at 0 Celsius for about 30 minutes. After 30 minutes, filter-off the precipitated product, and then vacuum dry or air-dry the crystals.

Note: Other salts of ecstasy (besides the hydrochloride) such as the sulfate, tartrate, citrate, and phosphate can be prepared by adding the corresponding acid to the ether mixture of the extracted freebase compound obtained at the end of the above process. For sulfuric acid or tartaric acid, 1 mole of 98% sulfuric acid or d-tartaric acid should be added to 2 moles of the ether mixture of the extracted freebase. For citric acid or phosphoric acid, 1 mole of the acid should be added to 3 moles of the ether mixture of the extracted freebase. The ether mixture after treatment with the corresponding acid in each of these cases can then be filtered to recover the precipitated crystals of the desired product. All the salts of ecstasy (other then the hydrochloride) are hallucinogens/stimulants and are psychedelic in nature. The tartrate and citrate salts may be twice as potent as the original hydrochloride (ecstasy).

0014. MDEA. Eve. N-ethyl-3,4-methylenedioxyphenylisopropylamine hydrochloride.

5-(2-methylpentyl)-1,3-benzodioxole hydrochloride



Structure | 3D model

MDEA is very similar to ecstasy in the areas of effects, duration, rates of onset, dosage, and duration of effects; however, Eve has demonstrated an "Alcohol like" intoxification when ingested by some users, with hallucinogenic or psychedelic effects not encountered by Ecstasy. Some of these hallucinogenic and/or psychedelic effects may include stupor, dizziness, and euphoria in combination with enhancements of sight, sound, smells, and touch. The stimulation effects of this drug are much less severe then those encountered in the amphetamines. A 200 milligram dose will produce a severe "stoned" effect in most people—this stoned effect is almost without any stimulation and ability to focus, speak properly, or coordinate. Currently the use of eve parallels that of ecstasy, and some brands of ecstasy, may actually include eve admixed. Data obtained on this compound indicates that it may have a slightly longer duration of effects in some people.

Note: This substance is a controlled substance (hallucinogen/stimulant) as listed in the US code of Federal regulations.

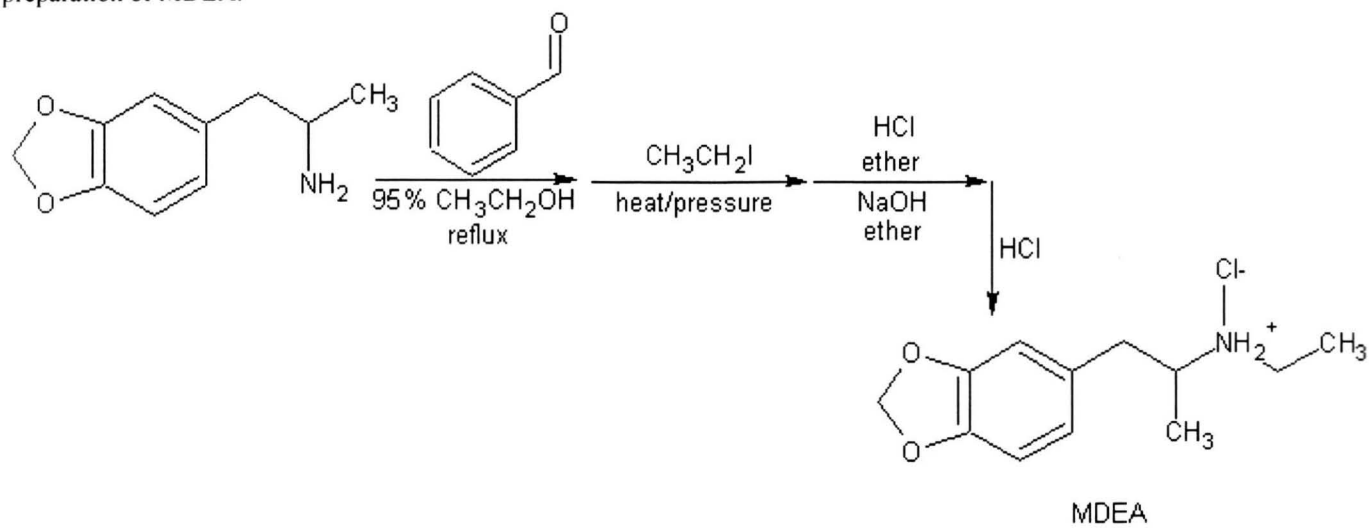
Toxicity: Low	**Rate of onset (average):** Rapid
Stimulation dosage (ingestion): 90 to 200 milligrams	**Duration of effects (average):** 4 to 7 hours (depending on the person)
Stimulation dosage (inhalation): 40 to 85 milligrams	**Habit forming potential:** Moderate
Stimulation dosage (injection): 30 milligrams +	**Estimated street value US (based on procedure):** $15 per gram

Procedure A: Preparation of MDEA

Materials:

1. 50 grams of MDA hydrochloride (see 0012. MDA hydrochloride)	6. 250 milliliters of 95% ethyl alcohol
2. 65 grams of sodium hydroxide	7. 31.2 grams of ethyl iodide
3. 750 milliliters of diethyl ether	8. 20 milliliters of methanol
4. 30 grams of anhydrous magnesium sulfate	9. 40 grams of 35 to 38% hydrochloric acid
5. 21.2 grams of benzaldehyde	10. 12 grams of dry hydrogen chloride gas

Summary: MDEA is prepared in an identical manner as for MDMA, but ethyl iodide is used in place of methyl iodide for the preparation of MDEA.



MDEA

Hazards: Extinguish all flames before using diethyl ether, which is highly flammable, and can form explosive mixtures with air. Use care when handling hydrogen chloride gas, which is very irritating to the nose and throat. 95% Ethyl alcohol, and methanol are both flammable, so keep away from fire. Note: methanol burns with a colorless flame, and burning methanol can be hard to see, especially outdoors.

Procedure:

Personnel notes for procedure A: MDEA

Place 50 grams of MDA hydrochloride into a beaker, and then add in a sodium hydroxide solution prepared by adding and dissolving 15 grams of sodium hydroxide into 50 milliliters of water. Note: sodium hydroxide generates much heat when dissolved in water, so allow the solution to cool to room temperature before using. After the addition of the sodium hydroxide, stir the entire mixture for 1 hour at ambient temperature (room temperature). Thereafter, extract the entire mixture with three-100 milliliter portions of diethyl ether, and after the extraction process, combine all ether portions (if not already done so), and then dry this combined ether portion by adding to it, 15 grams of anhydrous magnesium sulfate. Then stir the entire ether mixture for 10 minutes, and then filter-off the magnesium sulfate. Then place the filtered ether mixture into a distillation apparatus or rotary evaporator, and remove the ether. When no more ether distills over, recover the left over remaining oil residue (after it has cooled to room temperature), and then place 35.8 grams of this oil residue into a clean suitable reflux apparatus. Shortly thereafter, add in 21.2 grams of benzaldehyde, followed by 250 milliliters of 95% ethyl alcohol. Note: more ethyl alcohol may be needed to dissolve all the products—make sure all products are dissolved into the ethyl alcohol before proceeding. Thereafter, reflux the entire mixture for about 15 minutes at 78 Celsius. After the refluxing period, quickly remove the reflux condenser and replace it with a conventional condenser (fitted with a receiver flask), and then distill-off the ethyl alcohol. When no more ethyl alcohol passes over, stop the distillation process, and recover the left over remaining residue (after it has cooled). Now, place this left over residue into a suitable sized single neck flask, and then add in 31.2 grams of ethyl iodide. Thereafter, place a suitable sized balloon over the flask, and secure the balloon to the flask using a metal ring clamp.

Then heat the contents in the flask to about 100 Celsius for 5 hours. Note: the balloon will inflate and deflate sporadically during the heating process. The balloon is deigned to keep the contents of the flask under pressure to properly carryout the reaction. If during the heating process, the balloon pops or explodes, quickly replace with another one, and continue the operation for the necessary amount of remaining time. ***Note:*** *the pressure process just described, whereby a balloon is placed over a flask, can be substituted by using a conventional steel pipe with threads at both ends. To carryout the steel pipe technique, pour all necessary materials (as described for the balloon technique), into a thick walled stainless steel pipe, and then seal both ends with the corresponding steel caps. The threads at each end should be wrapped with Teflon tape prior to screwing in the end caps. Then place the entire pipe, and submerge it into a water bath and heat at the desired temperature for the desired time. Note: this process can be dangerous and can lead to pressure explosions. Carryout the process in an area that can contain any such explosion, and maintain a safe distance away during the operation—just to be on the safe side.* After the heating process, remove the heat source, and allow the reaction mixture to cool to room temperature. Note: monitor the balloon so it does not get sucked into the flask due to backpressure. Thereafter, remove the contents of the flask, and place them into a clean beaker. Then add in 20 milliliters of methanol, followed by 5 milliliters of water, and then stir the entire mixture for about 30 minutes. Then place this entire mixture into a clean reflux apparatus, and then reflux the entire mixture at 68 Celsius for 30 minutes. After refluxing for 30 minutes, quickly add in 100 milliliters of water, and then continue the reflux at 68 Celsius for 30 additional minutes. Thereafter, remove the heat source, and allow the refluxed mixture to cool to room temperature. Then pour the entire refluxed mixture into a clean suitable sized beaker, and then add in 40 grams of 35 to 38% hydrochloric acid, and then stir the entire mixture for about 30 minutes. Afterwards, quickly extract the entire mixture (to remove impurities) with three 50-milliliter portions of diethyl ether, and after the extraction process, the ether portions can be discard or recycled if desired. Now, to the extracted acidic mixture, add in a sodium hydroxide solution prepared by adding and dissolving 50 grams of sodium hydroxide into 190 milliliters of water. Note: sodium hydroxide generates much heat when dissolved in water, so allow the alkaline solution to cool to room temperature before using. Then stir the entire mixture for 30 minutes, and thereafter, extract the entire alkaline mixture with three 100-milliliter portions of diethyl ether. After the extraction process, combine all ether portions (if not already done so), and then wash this combined ether portion with three 100-milliliter portions of cold water. Note: during the extraction and washing, the ether will be the upper layer. After the washing period, dry the washed ether portion by adding to it, 15 grams of anhydrous magnesium sulfate, and then stir the whole mixture for about 10 minutes—thereafter, filter-off the magnesium sulfate. Finally, place the filtered ether mixture into an ice bath, and chill to 0 Celsius. Then bubble into the ether mixture, 12 grams of dry hydrogen chloride gas (an excess), and after the addition of the hydrogen chloride, filter-off the precipitated MDMA product, and then vacuum dry or air-dry the crystals.

Note: Other salts of eve (besides the hydrochloride) such as the sulfate, tartrate, citrate, and phosphate can be prepared by adding the corresponding acid to the ether mixture of the extracted freebase compound obtained at the end of the above procedure. For sulfuric acid or tartaric acid, 1 mole of 98% sulfuric acid or d-tartaric acid should be added to 2 moles of the ether mixture of the extracted freebase. For citric acid or phosphoric acid, 1 mole of the acid should be added to 3 moles of the ether mixture of the extracted freebase. The ether mixture after treatment with the corresponding acid in each of these cases can then be filtered to recover the precipitated crystals of the desired product. All the salts of eve (other then the hydrochloride) are hallucinogens/stimulants and are psychedelic in nature. The tartrate and citrate salts may be twice as potent as the original hydrochloride (eve).

Procedure B: Preparation of MDEA from piperonylacetone via amalgated aluminum reduction

Materials:

1. 40 grams of sodium hydroxide	8. 68 milliliters of a 25% sodium hydroxide solution
2. 20 grams of aluminum foil	9. 24.7 grams of piperonylacetone (see Intermediate-0011. Piperonylacetone)
3. 100 milliliters of 95% ethyl alcohol or 100 milliliters of denatured alcohol	10. 100 milliliters of methanol
4. 500 milligrams of mercury-II-chloride	11. 280 milliliters of 10% hydrochloric acid
5. 366 milliliters of diethyl ether	12. 495 milliliters of methylene chloride
6. 33.8 grams ethylamine hydrochloride	13. 15 grams of anhydrous magnesium sulfate
7. 247 milliliters of 99% isopropyl alcohol	14. 20 grams of hydrogen chloride gas

Summary: MDEA can be prepared by reacting piperonylacetone with amalgated aluminum in the presence of ethylamine hydrochloride. The reaction is identical to that in procedure B for the preparation of MDMA hydrochloride, and afterwards the reaction mixture is filtered, evaporated to remove solvents and water, and then extracted into hydrochloric acid, from where it forms the water-soluble hydrochloride. The hydrochloride is then purified by extraction of the freebase oil into methylene chloride by addition of sodium hydroxide, which liberates this freebase oil. The resulting methylene chloride mixture is then

evaporated to remove the methylene chloride, and the left over oil is then dissolved into ether, whereby it is finally precipitated as the purified MDEA.

Hazards: Extinguish all flames before using diethyl ether, which is highly flammable, and can form explosive mixtures with air. Wear gloves when handling mercury chloride, and any mercury containing solutions, or mixtures, as they can be absorbed into the skin. Ethyl alcohol, methanol, and isopropyl alcohol are flammable, and methanol burns with a colorless flame, so use caution. Sodium hydroxide, hydrochloric acid, and dry hydrogen chloride gas are corrosive, and capable of causing skin burns. Avoid inhalation of hydrogen chloride gas.

Procedure:

Personnel notes for procedure B: MDEA

Step 1: Amalgamation of aluminum

Into a suitable beaker, place 90 milliliters of distilled water, followed by 10 grams of sodium hydroxide. Thereafter stir the mixture to dissolve the sodium hydroxide. Note: much heat is generated when sodium hydroxide is dissolved in water, so allow the sodium hydroxide solution to cool to room temperature before using. Thereafter, add in 20 grams of aluminum foil pieces (cut into small squares), and allow the aluminum foil pieces to stand in the sodium hydroxide solution for about 20 minutes or until the evolution of hydrogen gas has drastically decreased. When the hydrogen gas evolution has almost ceased, filter-off the remaining pieces of aluminum, and then wash these collected pieces of aluminum with three 50-millilter portions of distilled water, followed by one portion of 50 milliliters of 95% ethyl alcohol (denatured alcohol can be used if desired). After the washing portion, allow the aluminum pieces to air-dry. When the pieces have air-dried, prepare a solution by adding and dissolving 500 milligrams of mercury-II-chloride (mercuric chloride) into 25 milliliters of water. Thereafter, add to the mercury chloride solution, the air-dried aluminum pieces, and allow the mixture to stand for about 15 minutes. After 15 minutes, filter-off the insoluble amalgated aluminum pieces, and then wash these filtered-off pieces with two 50-milliliter portions of distilled water, followed by one 50-milliliter portion of 95% ethyl alcohol (denatured alcohol will work if desired), and then wash with one portion of 10 milliliters of diethyl ether. After the washings, store the amalgated aluminum pieces submerged in a small amount of diethyl ether until use.

Step 2: Preparation of MDEA

Into a suitable flask or beaker, add in the amalgated aluminum prepared in step 1, followed by 33.8 grams of ethylamine hydrochloride dissolved in water (prepared by adding and dissolving the ethylamine hydrochloride into 30 milliliters of water), followed by 84 milliliters of 99% isopropyl alcohol, followed by 68 milliliters of a 25% sodium hydroxide solution, followed by 24.7 grams of piperonylacetone, and then followed by 163 milliliters of 99% isopropyl alcohol. Thereafter, moderately stir the reaction mixture for about 60 minutes. Note: during the reaction, keep the reaction mixtures temperature below 58 Celsius—a ice bath or cold water bath may or may not be needed, but most likely will be needed, so place the flask or beaker into a ice bath or ice water bath prior to adding the ingredients. After stirring the reaction mixture for 60 minutes, filter the reaction mixture to remove insoluble materials. Note: instead of filtering using the normal methods, pour a layer of celite (diatomaceous silicate powder) over the filter paper before filtering the reaction mixture after the initial 60-minute period. After filtering, pass two 50-milliliter portions of methanol through the filter (containing the celite), and then combine these two methanol portions to the filtered reaction mixture, and then place the entire reaction mixture into a distillation apparatus, and distill at 100 Celsius to remove the methanol, isopropyl alcohol, and water. When no more methanol, isopropyl alcohol, or water passes over or is collected, stop the distillation process, and allow the left over remaining oily residue to cool to room temperature before collecting it. Thereafter, dissolve the recovered oily residue into 56 milliliters of diethyl ether, and then extract this ether mixture with two 140-millilter portions of 10% hydrochloric acid. Note: after the extraction process, the hydrochloric acid mixture will be the lower layer each time. After the extraction process, briefly extract this hydrochloric acid mixture with three 25-milliliter portions of methylene chloride (to remove impurities), and then discard or recycle the methylene chloride portions. Note: after each extraction, the methylene chloride portion will be the lower layer each time. After the extraction process, basify the hydrochloric acid mixture by adding to it, a sodium hydroxide solution prepared by adding and dissolving 30 grams of sodium hydroxide into 150 milliliters of water. Note: sodium hydroxide generates much heat when dissolved in water, so allow the solution to cool to room temperature before using. After adding the sodium hydroxide solution, moderately stir the alkaline mixture for about 30 minutes at room temperature. Finally, extract this alkaline mixture with three 140-milliliter portions of methylene chloride, and after the extraction process, combine all methylene chloride portions (if not

already done so), and then dry this combined methylene chloride portion by adding to it, 15 grams of anhydrous magnesium sulfate. Then stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Then place this methylene chloride mixture into a distillation apparatus, and remove it at 40 Celsius. When no more methylene chloride passes over, recover the left over remaining oily residue (after it has cooled), and then dissolve it into 300 milliliters of diethyl ether. Thereafter, place this ether mixture into an ice bath, and chill to 0 Celsius. Then bubble into this chilled ether mixture, 20 grams (excess) of hydrogen chloride gas, and after the addition of the hydrogen chloride, stir the entire mixture for about 30 minutes at 0 Celsius. Then filter-off the precipitated MDEA product, and then vacuum dry or air-dry the crystals.

Note: Other salts of eve (besides the hydrochloride) such as the sulfate, tartrate, citrate, and phosphate can be prepared by adding the corresponding acid to the ether mixture of the extracted freebase compound obtained at the end of step 2. For sulfuric acid or tartaric acid, 1 mole of 98% sulfuric acid or d-tartaric acid should be added to 2 moles of the ether mixture of the extracted freebase. For citric acid or phosphoric acid, 1 mole of the acid should be added to 3 moles of the ether mixture of the extracted freebase. The ether mixture after treatment with the corresponding acid in each of these cases can then be filtered to recover the precipitated crystals of the desired product. All the salts of eve (other then the hydrochloride) are hallucinogens/stimulants and are psychedelic in nature. The tartrate and citrate salts may be twice as potent as the original hydrochloride (eve).

Procedure C: Preparation of MDEA

Materials:

1. 69 grams of sodium hydroxide	5. 60 milliliters of 35 to 38% hydrochloric acid
2. 70 grams of ethylamine hydrochloride	6. 375 milliliters of diethyl ether
3. 50 grams of 80% formic acid solution	7. 15 grams of anhydrous magnesium sulfate
4. 30 grams of piperonylacetone (see Intermediate-0011. Piperonylacetone)	8. 20 grams of dry hydrogen chloride gas

Summary: In this process, MDEA is formed in an identical manner as for procedure C for the preparation of MDMA; however, the methylamine is replaced with ethylamine. The MDEA is formed by the reaction of piperonylacetone with a 40% ethylamine solution in the presence of concentrated formic acid. The reaction mixture is heated at 160 Celsius for proper reaction temperature. After the initial reaction period, the reaction mixture is hydrolyzed with concentrated hydrochloric acid, and the reaction mixture is refluxed at 100 Celsius to properly carryout the hydrolysis. After the hydrolysis, the reaction mixture is diluted with water, and then briefly extracted with ether to dissolve impurities. The reaction mixture, after being extracted, is then basified by the addition of sodium hydroxide. After which, the alkaline reaction mixture is then extracted to dissolve the freebase oil. The combined ether portions are then treated with hydrogen chloride gas to precipitate the MDEA.

Hazards: Extinguish all flames before using diethyl ether, which is highly flammable and can form explosive mixtures with air. Concentrated formic acid, sodium hydroxide, and hydrochloric acid are capable of forming skin burns and irritation, so wear gloves when handling. Use proper ventilation when handling concentrated hydrochloric acid, which is a highly fuming substance.

Procedure:

Personnel notes for procedure C: MDEA

Into a suitable beaker, place a sodium hydroxide solution prepared by adding and dissolving 34 grams of sodium hydroxide into 40 milliliters of water. Note: sodium hydroxide generates much heat when dissolved in water, so allow the alkaline solution to cool to room temperature before using. Thereafter, slowly add in, 70 grams of ethylamine hydrochloride. During the addition, keep the temperature of the sodium hydroxide solution below 30 Celsius. After the addition of the ethylamine hydrochloride, stir the mixture for about 30 minutes at room temperature. Thereafter, filter the mixture to remove any insoluble solids. Then place this filtered mixture (which will contain a 40% solution of ethylamine in water) into a standard reflux apparatus with a 3-neck flask (the 3-neck flask should be equipped with thermometer, motorized stirrer, and addition funnel), followed by 50 grams of 80% formic acid solution. Thereafter, heat the contents in the 3-neck flask to about 160 Celsius, and when the temperature reaches 160 Celsius, add in all at once, 30 grams of piperonylacetone. Then reflux the entire reaction mixture at 160 Celsius for about 3.5 hours. During the reflux period, moderately stir the reaction mixture. After 3.5 hours,

reduce the heat to about 100 Celsius, and immediately after the temperature reaches 100 Celsius, place into the addition funnel, 60 milliliters of 35 to 38% hydrochloric acid (muriatic acid will work—31% hydrochloric acid), and then add this hydrochloric acid to the reaction mixture over a period of about 10 minutes. During the addition of the acid, continue to heat the reaction mixture at about 100 Celsius with moderate stirring. After 10 minutes, continue to reflux the reaction mixture at 100 Celsius under moderate stirring for 4 hours. After 4 hours, remove the heat source, and allow the reaction mixture to cool to room temperature. Thereafter, pour the entire reaction mixture into a suitable sized beaker, and then add in 100 milliliters of water. Thereafter, stir the entire reaction mixture for about 15 minutes. Then briefly extract the entire diluted reaction mixture with three 25-millilter portions of diethyl ether (to remove impurities), and after the extraction process, discard or recycle the ether portions. Note: after the extraction process, the ether will be the upper layer each time. Now, to the extracted reaction mixture, add in a sodium hydroxide solution prepared by adding and dissolving 35 grams of sodium hydroxide into 100 milliliters of water. Note: sodium hydroxide generates much heat when dissolved in water, so allow the sodium hydroxide solution to cool to room temperature before using it. After the addition of the sodium hydroxide solution, stir the entire alkaline mixture for about 30 minutes. Finally, extract this alkaline mixture with three 100-milliliter portions of diethyl ether, and then after the extraction process, combine all ether portions, if not already done so, and then wash this combined ether portion with three 50-milliliter portions of cold water. Note: after the extraction, and washings, the ether will be the upper layer each time. After the washing process, dry the washed ether portion by adding to it, 15 grams of anhydrous magnesium sulfate, and then stir the entire ether mixture for about 10 minutes—then filter-off the magnesium sulfate. Finally, place the filtered ether portion into an ice bath, and chill to 0 Celsius. Thereafter, bubble into the ether mixture, 20 grams of dry hydrogen chloride gas (an excess). After the addition of the hydrogen chloride gas, continue to stir the ether mixture for 1 hour, and then filter-off the precipitated MDEA product, and then vacuum dry or air-dry the crystals.

Note: Other salts of eve (besides the hydrochloride) such as the sulfate, tartrate, citrate, and phosphate can be prepared by adding the corresponding acid to the ether mixture of the extracted freebase compound obtained at the end of step 2. For sulfuric acid or tartaric acid, 1 mole of 98% sulfuric acid or d-tartaric acid should be added to 2 moles of the ether mixture of the extracted freebase. For citric acid or phosphoric acid, 1 mole of the acid should be added to 3 moles of the ether mixture of the extracted freebase. The ether mixture after treatment with the corresponding acid in each of these cases can then be filtered to recover the precipitated crystals of the desired product. All the salts of eve (other then the hydrochloride) are hallucinogens/stimulants and are psychedelic in nature. The tartrate and citrate salts may be twice as potent as the original hydrochloride (eve).

Procedure D: Preparation of MDEA directly from bromosafrole

Materials:

1. 24.5 grams of bromosafrole (see 0012. MDA hydrochloride, procedure A, step 1)	5. 350 milliliters of diethyl ether
2. 110 grams of a 40% ethylamine solution	6. 55 grams of sodium hydroxide
3. 50 milliliters of methanol	7. 15 grams of anhydrous magnesium sulfate
4. 100 milliliters of 35 to 38% hydrochloric acid	8. 25 grams of dry hydrogen chloride gas

Summary: MDEA is prepared in an identical manner as for the preparation of MDMA discussed in procedure D for MDMA. The reaction is quite simple and involves the amination of bromosafrole with ethylamine under heat and pressure. After the reaction, the contents of the reaction mixture are distilled to remove solvent, and the resulting solvent free mixture is then acidified, briefly extracted with ether (to remove impurities), and the resulting extracted mixture is then basified with sodium hydroxide to liberate the freebase oil. The freebase oil is then dissolved into ether by extraction, and the extracted ether mixture is then treated with hydrogen chloride gas to precipitate the MDEA.

Hazards: Extinguish all flames before using diethyl ether, which is highly flammable, and can form explosive mixtures with air. Use proper ventilation when handling ethylamine, which is a pungent irritating gas. Wear gloves when handling concentrated hydrochloric acid, and sodium hydroxide, both of which are capable of producing skin irritation.

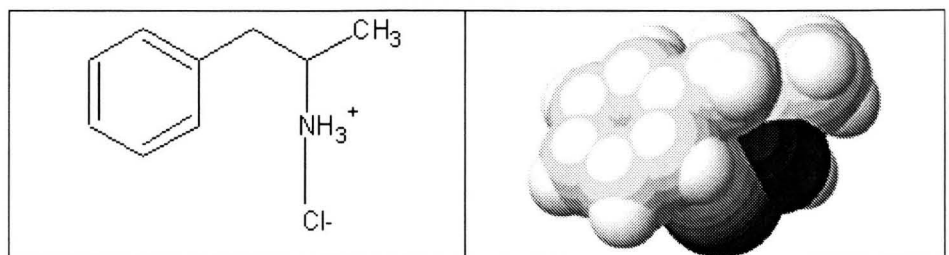
Procedure:

Personnel notes for procedure D: MDEA

Into a suitable beaker, place 24.5 grams of bromosafrole, followed by 110 grams of a 40% ethylamine solution. Thereafter, quickly add in 50 milliliters of methanol and then stir the entire mixture to ensure a uniform mixture. If after the addition of the methanol, two layers exist in the mixture, add a little more methanol until all layers disappear (merge). Thereafter, place the mixture into a suitable sized single neck flask, and then place a suitable sized balloon over the flask, and secure the balloon to the flask using a metal ring clamp. Then heat the contents in the flask to about 130 Celsius for 3 hours. Note: the balloon will inflate and deflate sporadically during the heating process. The balloon is deigned to keep the contents of the flask under pressure to properly carryout the reaction. If during the heating process, the balloon pops or explodes, quickly replace with another one, and continue the operation for the necessary amount of remaining time. ***Note:*** *the pressure process just described, whereby a balloon is placed over a flask, can be substituted by using a conventional steel pipe with threads at both ends. To carryout the steel pipe technique, pour all necessary materials (as described for the balloon technique), into a thick walled stainless steel pipe, and then seal both ends with the corresponding steel caps. The threads at each end should be wrapped with Teflon tape prior to screwing in the end caps. Then place the entire pipe, and submerge it into an oil bath and heat at the desired temperature for the desired time. Note: this process can be dangerous and can lead to pressure explosions. Carryout the process in an area that can contain any such explosion, and maintain a safe distance away during the operation—just to be on the safe side.* After the heating process, remove the heat source, and allow the reaction mixture to cool to room temperature. Then pour the entire reaction mixture into a distillation apparatus, and distill-off the methanol at 68 Celsius. When no more methanol distills over, stop the distillation process, and recover the left over remaining residue (after it has cooled to room temperature). Thereafter, place the left over remaining residue into a beaker, and then add in 100 milliliters of 35 to 38% hydrochloric acid (muriatic acid will work—31%), and then stir the entire acidic mixture for about 30 minutes. Then briefly extract the acidic mixture with two 25-milliliter portions of diethyl ether (to remove impurities), and after the extraction process, discard or recycle the ether portions. Thereafter, add to the extracted acidic mixture, a sodium hydroxide solution prepared by adding and dissolving 55 grams (excess) of sodium hydroxide into 150 milliliters of water. Note: sodium hydroxide generates much heat when dissolved in water, so allow the alkaline mixture to cool to room temperature before using. After the addition of the sodium hydroxide solution, stir the entire alkaline mixture for about 30 minutes. Finally, extract this alkaline mixture with three 100-milliliter portions of diethyl ether, and after the extraction process, combine all ether extracts (if not already done so), and then dry the combined ether portion by adding to it, 15 grams of anhydrous magnesium sulfate, and then stir the entire mixture for about 10 minutes. Thereafter, filter-off the magnesium sulfate, and then place the filtered ether mixture into an ice bath, and chill to about 0 Celsius. Then bubble into the mixture 25 grams of dry hydrogen chloride gas (excess), and after the addition, stir the entire mixture at 0 Celsius for about 30 minutes. After 30 minutes, filter-off the precipitated product, and then vacuum dry or air-dry the crystals.

Note: Other salts of eve (besides the hydrochloride) such as the sulfate, tartrate, citrate, and phosphate can be prepared by adding the corresponding acid to the ether mixture of the extracted freebase compound obtained at the end of the above process. For sulfuric acid or tartaric acid, 1 mole of 98% sulfuric acid or d-tartaric acid should be added to 2 moles of the ether mixture of the extracted freebase. For citric acid or phosphoric acid, 1 mole of the acid should be added to 3 moles of the ether mixture of the extracted freebase. The ether mixture after treatment with the corresponding acid in each of these cases can then be filtered to recover the precipitated crystals of the desired product. All the salts of eve (other then the hydrochloride) are hallucinogens/stimulants and are psychedelic in nature. The tartrate and citrate salts may be twice as potent as the original hydrochloride (eve).

0015. Amphetamine hydrochloride. *1-methyl-2-phenylethylamine hydrochloride*



Structure	3D model

Amphetamine hydrochloride forms crystals, which may be colorless to white, to yellow depending on purity. Freebase amphetamine is a mobile liquid, with an amine like odor. Amphetamine hydrochloride has several forms: the L and D forms are the most common, but a DL form is also encountered. Each form does give the amphetamine unique properties for each individual form, however, all forms of amphetamine have similar effects upon the body, so the exact forms will not be discussed in this book. The freebase has a boiling point of 203 Celsius, but can be distilled at 85 Celsius under a vacuum of 13 millimeters of mercury. Amphetamine hydrochloride is an old, and rather well know substance, and it is one of the first or original stimulants to be synthesized. Although amphetamine is not widely encountered as much as methamphetamine in the

drug trade, it is still a powerful stimulant with effects similar to methamphetamine; however, the effects of amphetamine are less then methamphetamine. Amphetamine is a powerful stimulant, which creates an extensive array of stimulations ranging from bursts of energy, to boosts in confidence and self-esteem, to heightened states of mental awareness. **Note: This substance is a controlled substance (stimulant) as listed in the US code of Federal regulations.**

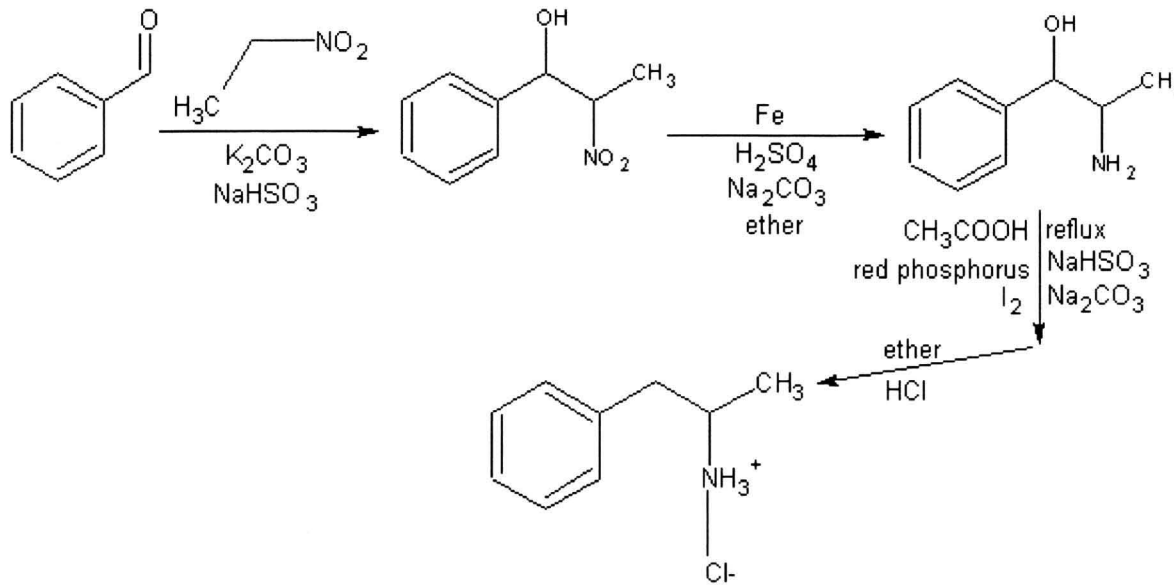
Toxicity: Low	**Rate of onset (average):** Rapid
Stimulation dosage (ingestion): 50 to 60 milligrams	**Duration of stimulation (average):** 5 to 8 hours (depending on the person)
Stimulation dosage (inhalation): 20 to 50 milligrams	**Habit forming potential:** High
Stimulation dosage (injection): 15 milligrams +	**Estimated value U.S. (based on procedure):** $21 per gram

Procedure A: Preparation of Amphetamine hydrochloride

Materials:

1. 53 grams of benzaldehyde	9. 80 grams of iron fillings
2. 37 grams of nitroethane	10. 300 grams of anhydrous sodium carbonate
3. 30 milliliters of a 30% potassium carbonate solution	11. 20 grams of dry hydrogen chloride gas
4. 950 milliliters of diethyl ether	12. 75 milliliters of glacial acetic acid
5. 90 milliliters of a 10% sodium bisulfite solution	13. 7.5 grams of red phosphorus
6. 60 grams of anhydrous magnesium sulfate	14. 16.7 grams of iodine crystals
7. 520 milliliters of 95% ethyl alcohol	15. 10 grams of sodium bisulfite
8. 533 grams of a 25% sulfuric acid solution	

Summary: Amphetamine can be made from phenylnitropropanol by reduction with iron and sulfuric acid, followed by reduction of the corresponding alcohol directly to amphetamine by reaction with iodine and red phosphorus in the presence of acetic acid. The desired amphetamine is recovered by basifying the reaction mixture, followed by extraction with ether. The ether mixture of the freebase is then treated with hydrogen chloride to precipitate the hydrochloride product. The phenylnitropropanol is obtained by condensing benzaldehyde with nitroethane. For similar or additional information, see serial number 433,816, March 6th, 1930 to Chogi Nagai of Shibuya Machi Japan, by Alexander Nagal, of Berlin Germany.



Hazards: Use maximum ventilation when handling diethyl ether and nitroethane, which are highly flammable and can form explosive mixtures with air. Wear gloves when handling nitroethane, iodine, and hydrogen chloride. Nitroethane can be absorbed through the skin, leading to potential poisoning. Iodine is a highly volatile solid, which readily forms a highly irritating vapor, so use proper ventilation when handling, and store iodine in amber glass bottles away from light.

Procedure:

Personnel notes for procedure A: Amphetamine hydrochloride

Step 1: Preparation of phenylnitropropanol

Into a suitable beaker or flask, place 53 grams of benzaldehyde, followed by 37 grams of nitroethane. Immediately thereafter, add in 30 milliliters of a 30% potassium carbonate solution, and rapidly stir the entire mixture at room temperature for 2 hours. Note: A cold-water bath may or may not be needed to keep the reaction mixture at ambient temperature (room temperature). Do not allow the reaction mixture to get above 25 Celsius. After stirring for 2 hours, add to the reaction mixture 200 milliliters of diethyl ether, and shortly thereafter, add in 90 milliliters of a 10% sodium bisulfite solution, and then moderately stir the entire reaction mixture for 30 minutes. Afterwards, place the entire reaction mixture into a seperatory funnel, and then remove the upper ether layer (after removing the lower aqueous layer). Thereafter, wash this upper ether layer with three 75-milliliter portions of cold water. Note: after each washing portion, use a seperatory funnel to recover the ether layer, which will be the upper layer each time. After the washing portion, add to the ether layer, 15 grams of anhydrous magnesium sulfate (to absorb water), and then stir the entire mixture for 10 minutes. Then filter-off the magnesium sulfate. Then, place the filtered ether mixture into a distillation apparatus, or rotary evaporator, and remove the ether. When no more ether passes over or is collected, remove the remaining oily residue (after allowing it to cool to room temperature), and then place aside for step 2. This oily residue will consist of the desired phenylnitropropanol.

Step 2: Preparation of 2-amino-1-phenylpropan-1-ol hydrochloride intermediate

Into a suitable 3-neck flask fitted with motorized stirrer, thermometer, and standard powder funnel, place 55 grams of the phenylnitropropanol obtained in step 1, and then add in 270 milliliters of 95% ethyl alcohol. Then briefly stir the entire mixture to form a uniform mixture. Thereafter, gradually add 533 grams of a 25% sulfuric acid solution and 80 grams of iron fillings to the phenylnitropropanol reaction mixture over a period of about 1 hour. Note: the sulfuric acid solution and iron fillings should be added in small portions at a time, one after the other, and this rate of addition should be continued for 1 hour. During the addition, rapidly stir the reaction mixture and maintain its temperature below 40 Celsius. After the additions are complete, continue to stir the reaction mixture for about 30 minutes at room temperature or below 40 Celsius. Then, add to the reaction mixture, 250 milliliters of 95% ethyl alcohol, and then stir the entire reaction mixture for about 30 minutes. Note: the addition of the alcohol at this point should cause ferrous sulfate to precipitate. After stirring the reaction mixture for 30 minutes, filter-off the precipitated ferrous sulfate. Now, place the filtered reaction mixture into a distillation apparatus or rotary evaporator, and remove the ethyl alcohol. When no more ethyl alcohol passes over or is collected, remove the remaining aqueous liquid (after it has cooled to room temperature), and place it into a suitable sized beaker. Then add in, a sodium carbonate solution prepared by adding and dissolving 100 grams of anhydrous sodium carbonate into 350 milliliters of water, and after the addition, rapidly stir the entire alkaline mixture for about 30 minutes at room temperature. Then extract the entire alkaline mixture with three 150-milliliter portions of diethyl ether, and after the extraction process combine all ether portions, if not already done so, and then dry this combined ether portion by adding to it, 25 grams of anhydrous magnesium sulfate. Note: during the extraction process, the ether will be the upper layer each time. After adding the anhydrous magnesium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Finally, place the filtered ether mixture into a suitable beaker, and then bubble into this mixture, 15 grams of dry hydrogen chloride gas (excess). After the addition of the hydrogen chloride gas, place the ether mixture into a distillation apparatus, and distil-off the ether only until 80% of the total volume has been removed. When 80% of the volume of the ether mixture has been removed, recover the left over remaining ether concentrate, after it has cooled to room temperature, and then filter-off the precipitated hydrochloride product. Then vacuum dry or air-dry the filtered-off crystals, and set aside for step 3.

Step 3: Preparation of amphetamine

Into a suitable flask, add 75 milliliters of glacial acetic acid, followed by 7.5 grams of red phosphorus, and then followed by 16.7 grams of iodine crystals. Thereafter, stir the entire mixture for about 15 minutes, and monitor the temperature to make sure it does not rise above 40 Celsius. After 15 minutes, much of the iodine will have reacted with the glacial acetic acid, if however, it appears a chemical reaction is still taking place after the initial 15 minutes, continue to stir the mixture for an additional 15 minutes. Thereafter, add in 25 milliliters of cold water, and then stir the entire mixture for about 10 minutes. Then, add in 3.7 grams of the 2-amino-1-phenylpropan-1-ol hydrochloride (obtained in step 2), and then stir the entire reaction mixture for about 30 minutes. Thereafter, pour the entire reaction mixture into a reflux apparatus, or simply attach a reflux condenser to the flask being used for the reaction, and then gently reflux (merely gently heat), the entire reaction mixture at 80 to 90 Celsius for about 90 minutes. Note: during the reflux operation (heating period), gently stir the reaction mixture. After the reflux period, remove the heat source, and allow the reaction mixture to cool to room temperature. Then add to it, 150 milliliters of cold water, and then stir the entire mixture for about 10 minutes, and then filter-off any insoluble materials. Now, gradually add to the filtered reaction mixture, a sodium bisulfite solution prepared by adding and dissolving about 10 grams of

sodium bisulfite into 500 milliliters of water. During the addition of this sodium bisulfite solution, rapidly stir the reaction mixture. After the addition of the sodium bisulfite solution (to destroy impurities), add in a sodium carbonate solution prepared by adding and dissolving 200 grams of anhydrous sodium carbonate into 750 milliliters of water. After the addition of the sodium carbonate solution, rapidly stir the entire reaction mixture for 1 hour at ambient temperature (room temperature). After 1 hour, extract the entire reaction mixture with four 75-milliliter portions of diethyl ether. After the extraction process, combine all ether extracts (if not already done so), and then dry this combined ether portion by adding to it 20 grams of anhydrous magnesium sulfate, and then stir the entire ether mixture for about 10 minutes. Thereafter, filter-off the magnesium sulfate, and then place this filtered ether mixture (containing the freebase of amphetamine) into an ice bath, and chill to about 0 Celsius. Thereafter, bubble into the chilled ether mixture, 5 grams of dry hydrogen chloride gas (excess). After the addition of the hydrogen chloride gas, filter-off the precipitated amphetamine hydrochloride product, and then vacuum dry or air-dry the product. Note: the filtered ether mixture can be evaporated to recover a small amount of additional product.

Note: Other salts of the freebase amphetamine such as the sulfate, tartrate, citrate, and phosphate can be prepared by adding the corresponding acid to the ether mixture of the amphetamine freebase compound obtained at the end of step 3. For sulfuric acid or tartaric acid, 1 gram of 98% sulfuric acid or 1.6 grams d-tartaric acid should be added to the ether mixture of the amphetamine freebase. For citric acid or phosphoric acid, 1.3 grams of citric acid or 700 milligrams of phosphoric acid should be added to the ether mixture of the amphetamine freebase. The ether mixture after treatment with the corresponding acid in each of these cases can then be evaporated using a distillation apparatus, or rotary evaporator to the point where only 80% of the total volume of the ether mixture has been reduced. The resulting ether concentrate can then be filtered to recover the product, which can then be vacuum dried or air-dried. All the salts of amphetamine are powerful stimulants, and the citrate salt may be more potent then the others. Note: if desired, the freebase amphetamine can be obtained by placing the freebase ether mixture obtained at the end of step 3, into a distillation apparatus and removing the ether. For purified freebase amphetamine, the amphetamine hydrochloride should be used, and treated with a 25% sodium carbonate (or sodium hydroxide) solution with stirring to liberate the freebase, which can then be extracted with ether, and the ether then removed to leave behind the freebase amphetamine.

Procedure B: Preparation of racemic-Amphetamine sulfate

Materials:

1. 27 grams of benzaldehyde	7. 68 milliliters of 35 to 38% hydrochloric acid
2. 20 grams of nitroethane	8. 22 grams of aluminum foil
3. 5 milliliters of cyclohexylamine	9. 200 milliliters of a 20% sodium hydroxide solution
4. 150 milliliters of dry hexane	10. 60 grams of sodium hydroxide
5. 250 milliliters of 95% ethyl alcohol	11. 100 milliliters dry acetone
6. 8 grams of nickel chloride hexahydrate	12. 4 grams of 98% sulfuric acid

Summary: Amphetamine sulfate can be made by reducing phenyl-2-nitropropene with a nickel salt. The nickel salt is prepared by reacting nickel powder with sodium hydroxide. The phenyl-2-nitropropene is prepared in the normal method by condensing benzaldehyde with nitroethane. The reaction of the phenyl-2-nitropropene with the nickel reducing agent is rather general, and pieces of aluminum, and portions of hydrochloric acid are added to compliment the reduction. After the phenyl-2-nitropropene has been reduced, the reaction mixture is then simply filtered, and the upper organic layer is then collected. This upper organic layer is then evaporated, and the left over oily residue is then dissolved into acetone, and the desired amphetamine sulfate is then precipitated by the addition of sulfuric acid.

Hazards: Extinguish all flames before using nitroethane, hexane, 95% ethyl alcohol, and acetone, as they are all highly flammable. Acetone and hexane are very volatile, so use proper ventilation when handling. Wear gloves when handling sodium hydroxide, sulfuric acid, and concentrated hydrochloric acid, as they are capable of causing skin irritation.

Procedure:

Personnel notes for procedure B: racemic-amphetamine sulfate

Step 1: Preparation of phenyl-2-nitropropene

Into a suitable reflux apparatus, place all at once, 27 grams of benzaldehyde, followed by 20 grams of nitroethane, followed by 5 milliliters of cyclohexylamine. Thereafter, reflux the entire mixture at about 100 Celsius for 3 hours. After refluxing for 3 hours, remove the heat source, and allow the two-phase reaction mixture to cool to room temperature. Then pour the entire reaction mixture into a seperatory funnel, and remove the lower (organic) layer. The upper layer can be recycled or discarded if desired as it will contain the cyclohexylamine catalyst. Then place the recovered lower organic layer into a suitable sized beaker, and then add in 25 milliliters of cold water. Immediately thereafter, rapidly stir the mixture using magnetic stirrer, or other means, for about 30 minutes at room temperature. Then remove the upper water layer by decanting it off, and then place the lower organic layer (containing the desired product), into an ice bath and chill to about 0 Celsius. Then, add in about 10 milliliters of cold water, and then allow the total mixture to stand at room temperature for several hours to allow the desired product of phenyl-2-nitropropene to crystallize. After 2 hours, most of the desired nitro compound should have precipitated and afterwards, filter-off the precipitated crystals, and then vacuum dry or air-dry them. Finally, recrystallize these dried collected crystals from 150 milliliters of dry hexane, and after the recrystallization process, vacuum dry or air-dry the crystals.

Step 2: Preparation of racemic-amphetamine sulfate

Into a suitable flask or beaker, place 150 milliliters of 95% ethyl alcohol, followed by about 8 grams of nickel chloride hexahydrate, and then heat the mixture to about 50 Celsius with stirring. When all the nickel chloride hexahydrate has dissolved, add in 3 milliliters of water, followed by 2 milliliters of 35 to 38% hydrochloric acid (muriatic acid will work), and continue stirring for an additional 5 minutes. Then, slowly add in, 10 grams of aluminum foil (regular old kitchen aluminum foil) in small portions at a time. During the addition of the aluminum foil pieces, moderately stir the reaction mixture. Note: the aluminum foil will only slowly react with the nickel chloride, so have patience. Continue stirring the reaction mixture for about 4 hours, or until the green color of the reaction mixture disappears. Note: stirring should be carried out with a glass stir rod or motorized stirrer, as nickel formation will stick to the magnetic stir bar. If after 4 hours, any green color still remains, add in another gram of aluminum foil until the green color has been discharged. When the green color of the reaction mixture has disappeared, stop stirring, and then filter-off the dark gray chunky precipitate (nickel), and then vacuum dry or air-dry the filtered-off precipitate. Then, place 200 milliliters of a 20% sodium hydroxide solution into a suitable flask or beaker, and then add in the dried filtered-off nickel precipitate in small portions over a period of about 10 minutes. During the addition, stir the sodium hydroxide solution. Thereafter, if the temperature of the reaction mixture has not risen to 60 Celsius, heat the reaction mixture to 60 Celsius with stirring. Then heat and stir the reaction mixture after the addition of the dried nickel precipitate for about 1 hour. The result will be a nickel solution containing a complex nickel salt, which is a strong reducing agent. Thereafter, filter-off the insoluble solids, and then discard them. Then place the nickel solution aside just for a moment. Now, place 10 grams of pheny-2-nitropropene (prepared in step 1) into a suitable flask or beaker, and then add in 100 milliliters of 95% ethyl alcohol, and then stir the entire mixture to dissolve all solids. Thereafter, add in the nickel solution over a period of about 5 minutes, and then immediately thereafter, slowly add in, 6 milliliters of 35 to 38% hydrochloric acid, followed by 2 grams of regular aluminum foil pieces. Then heat the reaction mixture to 60 Celsius. The addition of the hydrogen chloride and aluminum pieces should take only 10 to 15 minutes. Note: during both additions, moderately stir the reaction mixture. Note: a magnetic stirring bar should be omitted, and the reaction mixture should be stirred with a glass stir rod or motorized stirrer to prevent nickel from sticking to magnetic stir bar. After the addition of the hydrochloric acid and aluminum, stir and heat the reaction mixture at 60 Celsius until most of the aluminum dissolves. When most of the aluminum dissolves, slowly add in 60 milliliters of 35 to 38% hydrochloric acid, followed by an additional 10 grams of aluminum foil pieces. Both additions should only take 20 to 30 minutes. After all the additions, continue to stir the reaction mixture for 12 hours at 60 Celsius. After 12 hours, remove the heat source, and allow the reaction mixture to cool to room temperature. When it does, carefully add in, a sodium hydroxide solution prepared by adding and dissolving 60 grams of sodium hydroxide into 200 milliliters of water. Note: sodium hydroxide generates much heat when dissolved in water, so allow the solution to cool before using. After the addition of the sodium hydroxide solution, stir the entire mixture for about 1 hour using a glass stir rod or motorized stirrer. After 1 hour, filter the entire two-phase mixture (to remove precipitated nickel and aluminum sludge), and then place the entire filtered two-phase mixture into a seperatory funnel, and remove the upper ethyl alcohol layer (orange layer), after removing the lower aqueous layer. Thereafter, place the upper ethyl alcohol layer into a distillation apparatus, and distill-off the ethyl alcohol at 78 Celsius. When no more ethyl alcohol passes over or is collected, stop the distillation process, and remove the left over remaining oily residue (after is has cooled). Finally, dissolve the left over remaining oily residue into 100 milliliters of dry acetone, and then quickly filter this acetone mixture to remove any insoluble impurities. Then place this filtered acetone mixture into an ice bath and chill to 0 Celsius. Thereafter, slowly add to this filtered acetone mixture, 4 grams of 98% sulfuric acid. Then stir the entire acetone mixture for about 30 minutes at 0 Celsius, and then filter-off the precipitated crystals, and then vacuum dry or air-dry the crystals. The result will be about 6 grams of the desired racemic-amphetamine sulfate.

Procedure C: Preparation of racemic-Amphetamine hydrochloride from toluene

Materials:

1. 150 milliliters of dry toluene	10. 125 milliliters of dry methyl alcohol
2. 30 grams of chlorine gas	11. 7.9 grams of sodium borohydride

3. 5 grams of finely divided sodium cyanide	12. 30 grams of 35 to 38% hydrochloric acid
4. 15 grams of 95% ethyl alcohol	13. 260 milliliters of methylene chloride
5. 50 milliliters of 95% ethyl alcohol	14. 10 grams of sodium hydroxide
6. 3 milliliters of 98% sulfuric acid	15. 10 grams of anhydrous sodium sulfate
7. 12.5 grams of dry methyl iodide	16. 25 milliliters of a 5% sodium bicarbonate solution
8. 295 milliliters of dry tetrahydrofuran (THF)	17. 100 milliliters of diethyl ether
9. 25 grams of magnesium turnings	18. 10 grams of hydrogen chloride gas

Summary: *Note: This process can be used to mass-produce amphetamine from readily available toluene, and small pilot plants can be permanently set-up for this entire process.* Amphetamine can be produced in an indirect method, starting from toluene in a three-step process. The first step involves the formation of benzyl chloride, which is prepared by the photo chlorination of toluene at 120 Celsius. After the reaction, the desired benzyl chloride can be obtained by distillation using a multiple path distillation apparatus. Multiple path distillation apparatus takes advantage of density, and each fraction will carry over into its own receiver flask based on its density. Because benzyl chloride is more dense then toluene, but less dense then benzyldichloride, it collects in the second receiver flask. After the distillation process, the benzyl chloride can be purified by fractional distillation. Note: benzotrichloride will also be a by-product, and it will remain in the distillation flask. Once the benzyl chloride has been prepared, and has been collected, the rest of the process is relatively down hill. The second step involves the formation of benzyl cyanide, which is conveniently prepared from the benzyl chloride by reaction with sodium cyanide in the presence of alcohol. The reaction is rather general, and after refluxing the reaction mixture for a short amount of time, the reaction mixture is filtered, and then distilled to remove water and solvent. The left over liquid is then treated with a semi-concentrated sulfuric acid solution to break down impurities. Afterwards, the benzyl cyanide layer is removed from the aqueous acid by use of a seperatory funnel, and then distilled using a two-path distillation apparatus—whereby the desired benzyl cyanide collects in the upper receiver flask. This benzyl cyanide fraction is then converted into amphetamine hydrochloride by reaction with methyl magnesium iodide in the presence of tetrahydrofuran. The reaction produces an interesting magnesium containing intermediate, which is broken down into the freebase amphetamine by reduction with sodium borohydride. The methylmagnesium iodide is generated by the reaction of methyl iodide with magnesium turnings. After reduction with the sodium borohydride, the reaction mixture is concentrated by evaporation, and the left over residue is then taken-up into water, acidified, and then extracted with solvent to remove impurities. Thereafter, the extracted acidified aqueous mixture is then extracted with solvent, the solvent portions are combined, dried, and then evaporated in the usual manner. Thereafter, the left over oil thus obtained is then dissolved into ether, and the amphetamine hydrochloride is precipitated by the addition of hydrogen chloride gas.

Hazards: Use proper ventilation when using diethyl ether and tetrahydrofuran, as they are both highly flammable and capable of forming explosive mixtures with air. Wear gloves when handling sodium cyanide, and avoid contact with the skin. Keep sodium cyanide away from acids at all times. Wear gloves when handling sodium hydroxide, sulfuric acid, and hydrochloric acid, as they are all capable of producing mild irritation. Magnesium turnings are flammable, so keep them sway from strong sources of ignition. Methyl alcohol burns with a colorless flame, so burning methyl alcohol can be hard to see; keep methyl alcohol away from fire and other sources of ignition. Wear gloves when handling sodium borohydride, and it keep it out of contact with water. Use proper ventilation when handling toluene, and avoid inhalation of the vapors.

Procedure:

Personnel notes for procedure C: racemic-amphetamine hydrochloride

Step 1: Preparation of benzyl chloride through chlorination of toluene

Into the apparatus as illustrated below, place 150 milliliters of dry toluene, followed by a few glass beads (about 2 to 4 millimeters in diameter each glass bead). Thereafter, reflux the toluene at about 110 to 120 Celsius. Note: the temperature should be slowly brought up at the start of the reflux—no rapid heating. When the toluene reaches a temperature of about 90 to 100 Celsius, start bubbling in the chlorine gas (about 30 grams of chlorine gas total; an excess), over a period of about several hours—the chlorine gas addition should not be to fast, and only a nice slow steady stream of gas is desired. Note: the chlorine gas should also be as dry as possible. During the chlorine gas addition, monitor the temperature of the toluene, and do not let it rise above 140 to 150 Celsius. Also, during the addition of the chlorine gas, rapidly stir the toluene. **Note: this entire reaction should be carried out by exposing the reaction flask of the apparatus to direct sunlight, UV light from any suitable UV**

light bulb, mercury vapor lamp, or halogen lamp. To do this, simply expose the apparatus (preferably only the reaction flask containing the toluene) to direct sunlight (by placing it outside on a sunny day, placing it on a window ledge with the window open on a sunny day, ect., ect.,), or simply place the corresponding light bulb 12 to 18 inches away from the reaction flask containing the toluene. A powerful regular light bulb, say a 180 to 220 watt light bulb can also be used to speed up the reaction, but only by 25 to 30% of the ability of sunlight or other UV light sources. To use a regular 180 to 220 watt light bulb, simply place the light bulb 1 to 2 inches away from the reaction flask containing the toluene. If not using direct sunlight or any other UV light source, the reaction will take much longer, so continue to reflux the toluene at about 110 to 120 Celsius for about 8 to 10 hours (even if the chlorine addition has been completed for some time). If using direct sunlight, or another UV light source, continue to reflux the reaction mixture at about 110 to 120 Celsius for about 3 hours (even if the chlorine addition has been completed for some time). Note: if using a standard light bulb (as of 180 to 220 watt), continue to reflux the toluene at 110 to 120 Celsius for about 5 to 6 hours (even if the chlorine addition has been completed for some time). After the necessary amount of time for refluxing the toluene mixture as ended, stop the reflux process, and allow the toluene reaction mixture to cool to room temperature.

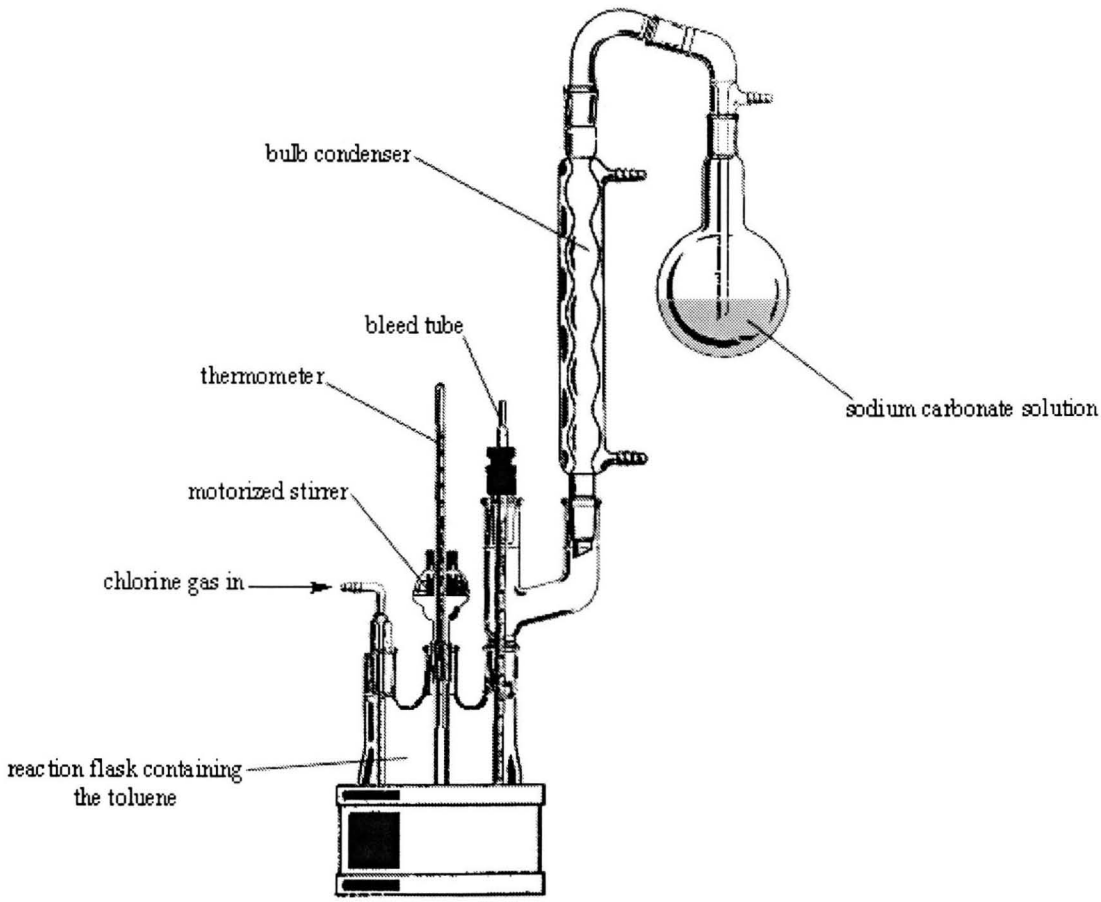


Figure 044. Set-up for the preparation of benzyl chloride. Employed here is a 4-neck reaction flask (the fourth neck is concealed—set behind the center neck). The bleed tube should be submerged just below the surface of the boiling toluene, and is designed to act as relief for any back-pressure in the apparatus. The sodium carbonate solution will neutralize any hydrogen chloride gas formed as a byproduct during the reaction. The chlorine gas inlet tube should be submerged below the surface of the boiling toluene.

After the toluene reaction mixture has cooled to room temperature, pour it into a multiple path distillation apparatus (as illustrated below). Then distill this toluene reaction mixture at about 180 Celsius. Note: if a heating mantle or hot plate capable of achieving such a high temperature is unavailable, use a Bunsen burner—but monitor the flame, and the temperature of the toluene reaction mixture carefully—do not exceed 200 Celsius. Note: obviously, vacuum distillation would be more convenient, and is recommend if such technology is available.

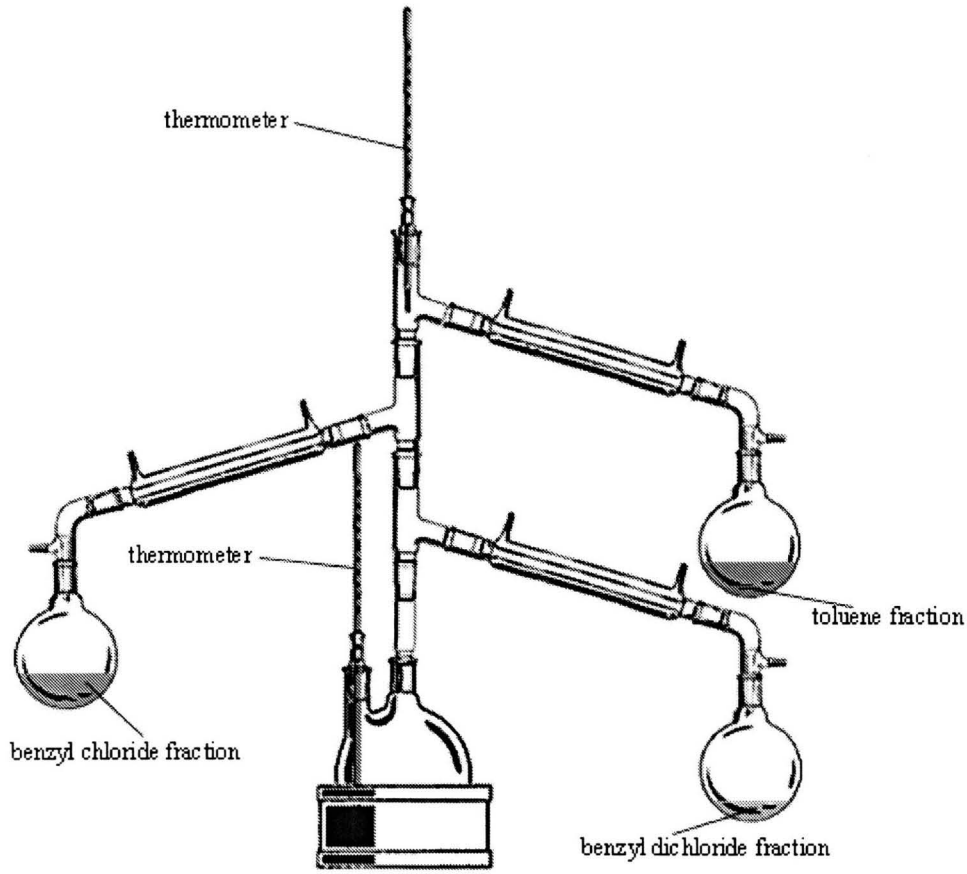


Figure 045. Multiple path distillation apparatus for the distillation of benzyl chloride. Note: the benzyl chloride fraction should then be re-distilled at 179 Celsius using a fractional distillation apparatus.

Step 2: Preparation of benzyl cyanide

Into a suitable sized reflux apparatus, equipped with addition funnel, thermometer, and motorized stirrer or other stirring means, place 5 grams of finely divided sodium cyanide, followed by 10 milliliters of warm water. Thereafter, briefly stir the entire mixture to form a uniform mix. Then prepare a solution by adding and dissolving 10 grams of benzyl chloride (prepared in step 1) into 15 grams of 95% ethyl alcohol. Thereafter, place this benzyl chloride mixture into the addition funnel, and then slowly add it, drop-wise to the sodium cyanide mixture over a period of time sufficient to keep the sodium cyanide mixture below 40 Celsius. During the addition of the benzyl chloride mixture, rapidly stir the sodium cyanide mixture. Note: the addition should take no longer then 10 to 15 minutes. After the addition of the benzyl chloride/alcohol mixture, reflux the entire reaction mixture at 79 celsius for about 1 hour with rapid stirring After refluxing the entire reaction mixture for about 1 hour, stop the reflux process, and then allow the reaction mixture to cool to room temperature. Thereafter, filter the reaction mixture to remove insoluble sodium chloride, and then wash this filtered-off sodium chloride with two 25-milliter portions of 95% ethyl alcohol. Then combine both of these washing portions with the filtered reaction mixture, and then place this combined filtered reaction mixture into a distillation apparatus (equipped with motorized stirrer or other stirring means). Now, before distilling the mixture, add in 20 milliliters of water, and then distill the entire reaction mixture at 100 Celsius to remove water and alcohol. Note: during the distillation process, rapidly stir the entire reaction mixture. When no more water or alcohol passes over or is collected, stop the distillation process, and then allow the remaining left over contents to cool to room temperature. Thereafter, filter this left over remaining liquid (to remove any impurities; if any). Now, place this filtered left over liquid into a clean flask or beaker, and then add in a warm sulfuric acid solution prepared by adding and mixing 3 milliliters of 98% sulfuric acid into 5 milliliters of water. Note: sulfuric acid generates excessive heat when dissolved in water, so allow the acid solution to cool to about 50 to 60 Celsius before using. After adding in the warm acid solution, rapidly stir the entire mixture for about 30 minutes. After 30 minutes, place the entire mixture into a seperatory funnel, and then remove the upper benzyl cyanide layer. Then place this upper benzyl cyanide layer into a clean beaker, and then add in, 25 milliliters of a 5% sodium bicarbonate solution, and then stir the entire mixture for about 10 minutes. Thereafter, place this entire two-phase mixture into a seperatory funnel, and remove the lower benzyl cyanide layer. Note: in some cases, the benzyl cyanide might be the upper layer. Finally, place this benzyl cyanide layer into a two-path distillation apparatus (similar to the multiple path distillation apparatus used in step 1, except with only two paths, not three), and then distill the benzyl cyanide at 234 Celsius.

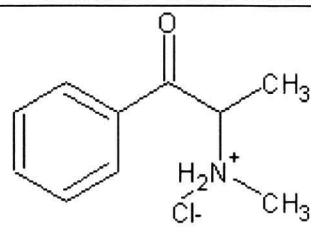
Note: if a high temperature heating mantle or hot plate is un-available, a Bunsen burner can be used. If using a Bunsen burner, monitor the flame and temperature of the benzyl cyanide closely—do not exceed 250 Celsius. Obviously, vacuum distillation works best. Second note: the benzyl cyanide fraction will collect in the highest receiver flask (the second path). After the distillation process, collect the benzyl cyanide fraction, and then set it aside for step 3. Note: prior to using this benzyl cyanide (for step 3), add to it, a small amount of anhydrous sodium sulfate and then stir the entire mixture for about 5 minutes—thereafter, filter-off the sodium sulfate. This process is to make sure the benzyl cyanide is dry before using it in step 3.

Step 3: Preparation of amphetamine hydrochloride

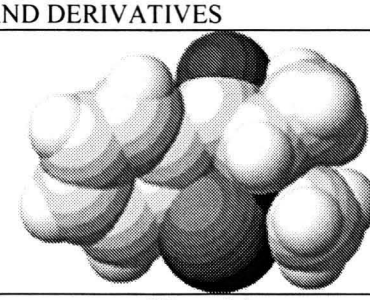
Into a standard reflux apparatus, equipped with motorized stirrer or other stirring means, addition funnel, and thermometer, place 12.5 grams of dry methyl iodide, followed by 170 milliliters of dry tetrahydrofuran. Note: place a calcium chloride drying tube over the reflux condenser to exclude moisture. Thereafter, place this mixture into an ice bath, and chill to about 0 Celsius. Thereafter, slowly add, in small portions at a time, 25 grams of magnesium turnings (through the top of the reflux condenser—temporarily remove the calcium chloride drying tube). During the addition of the magnesium turnings, slowly stir the methyl iodide solution, and maintain its temperature at 0 Celsius at all times. After the addition of the magnesium turnings, continue to slowly stir the reaction mixture for about 10 additional minutes. Now, into a separate clean reflux apparatus, equipped with motorized stirrer or other stirring means, and addition funnel, place 125 milliliters of dry tetrahydrofuran, followed by 9.7 grams of dry benzyl cyanide (prepared in step 2). Then place this benzyl cyanide mixture into a cold-water bath, and chill to about 15 Celsius. Thereafter, stir this entire mixture briefly to form a uniform mix. Thereafter, place the reaction mixture (of the methyl iodide and magnesium, just previously prepared), into the addition funnel attached to the new reflux apparatus (containing this benzyl cyanide mixture), and then slowly add the methyl iodide/magnesium reaction mixture to the benzyl cyanide mixture over a period of time sufficient to keep the temperature of the benzyl cyanide mixture around 20 to 25 celsius. During the addition, rapidly stir the benzyl cyanide mixture. After the addition, continue to stir the entire new reaction mixture for about 90 minutes at room temperature, and thereafter, remove the cold-water bath, and replace it with an ice bath. Then chill this new reaction mixture to 0 Celsius. Thereafter, add in 125 milliliters of dry methyl alcohol, and then stir the entire new reaction mixture for about 5 minutes. Then slowly add in, in small portions at a time, 7.9 grams of sodium borohydride over a period of about 30 minutes. During the addition of the sodium borohydride, rapidly stir the new reaction mixture, and maintain its temperature below 5 Celsius at all times. After the addition of the sodium borohydride, continue to stir the new reaction mixture at a temperature below 5 Celsius, for an additional 50 minutes. Afterwards, place this entire new reaction mixture into a distillation apparatus, and distill-off the tetrahydrofuran at 66 Celsius. When no more tetrahydrofuran passes over or is collected, stop the distillation process, and recover the left over resinous material (after it has cooled). Then place this left over resinous material into a clean beaker, and then add in 170 milliliters of warm water, and then stir the entire mixture for about 30 minutes. Now, add in, 30 grams of 35 to 38% hydrochloric acid (muriatic acid of 31% will work), and then rapidly stir the entire acidic aqueous mixture for about 30 minutes. After 30 minutes, briefly extract this acidic aqueous mixture with two 40-milliter portions of methylene chloride (to remove impurities), and after this brief extraction, the methylene chloride portions can be recycled or discarded if desired. Note: during this brief extraction process, the methylene chloride will be the lower each time. After the brief extraction process, place the collected acidic aqueous mixture into a suitable sized beaker, and then add in a sodium hydroxide solution prepared by adding and dissolving 10 grams of sodium hydroxide into 50 milliliters of water. Note: sodium hydroxide generates excessive heat when dissolved in water, so allow the alkaline solution to cool before using. After adding in the sodium hydroxide solution, rapidly stir the now entire alkaline mixture for about 30 minutes. Thereafter, extract this alkaline mixture with three 60-milliter portions of methylene chloride, and after the extraction process, combine all methylene chloride portions (if not already done so), and then dry this combined methylene chloride portion by adding to it, 10 grams of anhydrous sodium sulfate. Note: after each extraction process the methylene chloride will be the lower each time. After adding in the anhydrous sodium sulfate, stir the entire methylene chloride portion for about 10 minutes, and then filter-off the sodium sulfate. Finally, place this dried filtered methylene chloride portion into a distillation apparatus, and distill-off the methylene chloride. When no more methylene chloride passes over or is collected, stop the distillation process, and recover the left over remaining oil (after it has cooled). Then dissolve this oil into 75 milliliters of diethyl ether, and then stir this entire mixture for about 10 minutes. Thereafter, filter this ether mixture to remove any insoluble materials (if any), and then place this filtered ether mixture into an ice bath. Thereafter, bubble into this ether mixture, 10 grams of hydrogen chloride gas, and after the addition, allow this ether mixture to stand at 0 Celsius for about 1 hour. After 1 hour, filter-off the precipitated crystals of the amphetamine hydrochloride, wash them with one portion of 25 milliliters of diethyl ether, and then vacuum dry or air-dry the crystals.

Note: other salts of the freebase amphetamine can be prepared by replacing the hydrogen chloride gas (used at the end of the above process) with the corresponding acid, in the usual manner.

0016. CAT. Methcathinone. *2-methyl-1-phenylbutan-1-one hydrochloride*



Structure



3D model

CAT forms an off-white, to white, to slightly yellow solid, powder, or granules, with a melting point of 170 to 182 Celsius (depending on purity. CAT like many other drugs, has several forms ranging form the L-form, to the D-form, to the DL-form. However, the importance of these various forms, although meaningful to the medial community, has no real meaning when applied to street use—as any form will work as a stimulant/psychedelic with satisfactory results. CAT is a very simple, yet interesting methamphetamine derivative with powerful analeptic activity. CAT is relatively unknown to most people, but its demand will definitely increase with time. Persons describing its effects upon the body have stated it as being "spectacular", and even much better then cocaine or methamphetamine. Its stimulation effects are similar to cocaine and methamphetamine, but it appears in addition, that CAT also demonstrates interesting "upper" effects other then CNS stimulation, with secondary psychedelic effects. Users of the drug have stated it to produce a brilliant psychological experience producing heavy bursts of energy, overwhelming happiness, confidence, and self-esteem, total elimination of fear, or nervousness, and pin-point mental awareness, with excellent reflex action. Secondary effects include hallucinogenic, and/or psychedelic effects with strange physical enhancements (to the body—sight, smell, touch, ect., ect.,) being experienced with lights and lighting, colors, and even weather---including an increased enhancement of rain, snow, and even fog.

Note: This substance is a controlled substance (stimulant/psychedelic) as listed in the US code of Federal regulations.

Toxicity: Very low	**Rate of onset (average):** Rapid
Stimulation dosage (ingestion): 500 to 900 milligrams	**Duration of effects (average):** 8 to 12 hours, but some people may feel effects for up to 6 days (depending on the person)
Stimulation dosage (inhalation): 300 to 350 milligrams	**Habit forming potential:** Very high
Stimulation dosage (injection): 150 milligrams +	**Estimated value U.S. (based on procedure):** $18 per gram

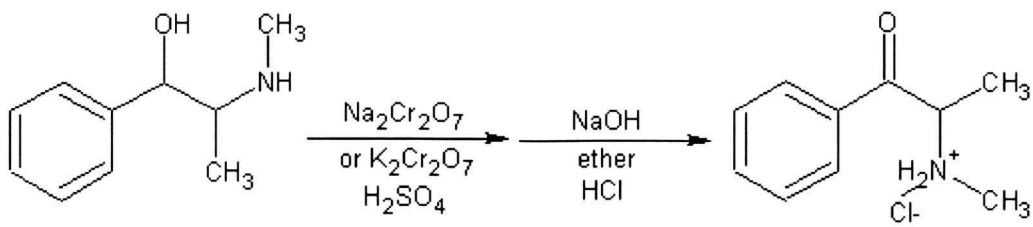
Procedure A: Preparation of CAT

Materials:

1. 8.25 grams of ephedrine (freebase) (see Intermeidate-0007. Ephedrine) or pseudoephedrine	6. 100 milliliters of methylene chloride
2. 9.2 grams of 98% sulfuric acid	7. 100 milliliters of diethyl ether
3. 5 grams of sodium dichromate	8. 15 grams of anhydrous magnesium sulfate
4. or 5.6 grams of potassium dichromate	9. 5 grams of dry hydrogen chloride gas
5. 40 grams of sodium hydroxide	

Summary: CAT is readily prepared by the oxidation of ephedrine or pseudoephedrine with sodium or potassium dichromate in the presence of sulfuric acid. The reaction is rather general, and the desired product is readily obtained by basifying the reaction mixture, and then extraction with methylene chloride followed by ether. The solvent extracts are then combined, dried, treated with hydrogen chloride, and then evaporated. The evaporated mixture is then filtered to recover the crystals of the hydrochloride. For additional information, see serial number 437,012, June 15th, 1954 to Yvon J. L'italien, of Hazel Park Mi, and Mildred C. Rebstock, of Detroit Mi, assigned by Parke, Davis & Company.

Note: for the following process, if L-ephedrine is used, the CAT will be the DL form.



ephedrine

CAT

Hazards: Extinguish all flames before using diethyl ether, which is highly flammable, and can form explosive mixtures with air. Wear gloves when handling concentrated sulfuric acid and sodium hydroxide, both of which are capable of producing skin burns. Potassium and sodium dichromate are strong oxidizers, so keep them away form combustible materials.

Procedure:

Personnel notes for procedure A: CAT

Into a suitable beaker, equipped with magnetic stirrer bar, place 8.25 grams of freebase ephedrine (any form will do), followed by 25 milliliters of water, followed by 2.6 grams of 98% sulfuric acid. Thereafter, stir the contents in the beaker until they reach a temperature of room temperature (as sulfuric acid generates heat when added to water). Thereafter, prepare a solution in a separate beaker, by adding in 5 grams of sodium dichromate or 5.6 grams of potassium dichromate, followed by 6.6 grams of 98% sulfuric acid, followed by 23 milliliters of cold water. Thereafter, stir this solution for about 30 minutes until all solids have dissolved, and then allow this solution to cool to room temperature before using (as sulfuric acid generates heat when dissolved in water). Then, add this sodium dichromate or potassium dichromate solution to the ephedrine solution, over a period sufficient to keep the ephedrine mixture (reaction mixture) below 40 Celsius at all times. During the addition, rapidly stir the reaction mixture. After the addition, continue to stir the reaction mixture for 12 hours at room temperature. After 12 hours, add to the reaction mixture, a sodium hydroxide solution made by dissolving 40 grams of sodium hydroxide into 175 milliliters of water. Note: sodium hydroxide generates much heat when dissolved in water, so allow the sodium hydroxide solution to cool to room temperature before using. During the addition of the sodium hydroxide, moderately stir the reaction mixture. After the addition of the sodium hydroxide solution, extract the entire alkaline mixture with two 50-milliliter portions of methylene chloride, followed by two 50-millilter portions of diethyl ether. After the extraction process, combine all methylene chloride and ether extracts (to each other), if not already done so, and then dry this combined methylene chloride/ether portion by adding to it, 15 grams of anhydrous magnesium sulfate. Then stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Finally, bubble into this combined solvent portion, 5 grams of dry hydrogen chloride gas. After the addition of the hydrogen chloride gas, place the entire mixture into a distillation apparatus, and distill-off the methylene chloride and ether at 40 Celsius, only until 80% of the total volume has been removed. When this point is reached, stop the distillation, and recover the left over remaining solvent concentrate (after it has cooled to room temperature), and then filter-off the insoluble solids, which will the desired product as the hydrochloride. These crystals can then be vacuum dried or air-dried. To purify, recrystallize the desired CAT from ethyl alcohol (1 gram CAT per 10 milliliters of 95% ethyl alcohol, and then add in ether—1 part ether per 1 part 95% ethyl alcohol, to precipitate the purified CAT, which can then be collected by filtration, and then vacuum dried or air-dried.

Note: Other salts of the freebase CAT such as the sulfate, tartrate, citrate, and phosphate can be prepared by adding the corresponding acid to the methylene chloride/ether mixture of the freebase CAT compound obtained at the end of the above process. For sulfuric acid or tartaric acid, 1 mole of 98% sulfuric acid or d-tartaric acid should be added to 2 moles of freebase CAT in the methylene chloride/ether mixture. For citric acid or phosphoric acid, 1 mole of citric acid or phosphoric acid should be added to the freebase CAT in the methylene chloride/ether mixture. The methylene chloride/ether mixture after treatment with the corresponding acid in each of these cases is then evaporated using a distillation apparatus, or rotary evaporator to the point where only 80% of the total volume of the methylene chloride/ether mixture has been reduced. The resulting solvent concentrate can then be filtered to recover the product, which can then be vacuum dried or air-dried. All the salts of CAT are powerful stimulants/psychedelics, and the citrate salt may be more potent then the others. Note: if desired, the freebase CAT can be obtained by placing the freebase CAT in the methylene chloride/ether mixture obtained at the end of the above process, into a distillation apparatus and removing the methylene chloride/ether until no more solvent is collected, and then recovering the left over residue of CAT (after it has cooled). For purified freebase CAT, the regular CAT should be used, and treated with a 25% sodium carbonate (or sodium hydroxide) solution with stirring to liberate the freebase CAT, which can then be extracted with methylene chloride/ether (as in the procedure), and the solvent mixture then removed to leave behind the purified freebase CAT.

Procedure B: Preparation of CAT using potassium permanganate

Materials:

1. 10 grams of ephedrine (freebase) (see Intermeidate-0007. Ephedrine)	7. 900 milliliters of methylene chloride

2. 75 milliliters of diethyl ether	8. 90 grams of sodium hydroxide
3. 25 grams of dry hydrogen chloride gas	9. 300 grams of a 5% sulfuric acid solution
4. 45 milliliters of glacial acetic acid	10. 25 grams of anhydrous magnesium sulfate
5. 6 grams of potassium permanganate	11. 100 milliliters of hexane
6. 15 grams of sodium bisulfite	

Summary: CAT is readily prepared in a similar manner as for procedure A., but potassium permanganate in the presence of glacial acetic acid is the oxidizer of choice to convert ephedrine hydrochloride into the desired CAT. The reaction is rather mild, and the desired CAT is recovered in the usual manner.

Hazards: Extinguish all flames before using diethyl ether, which is highly flammable, and can form explosive mixtures with air. Hexane is also very flammable, so keep any source of ignition away at all times when using. Wear gloves when handling sodium hydroxide, glacial acetic acid, and hydrogen chloride gas, all three of which are capable of producing skin burns and/or skin irritation. Potassium permanganate is a powerful oxidizer, so keep out of contact with combustible materials.

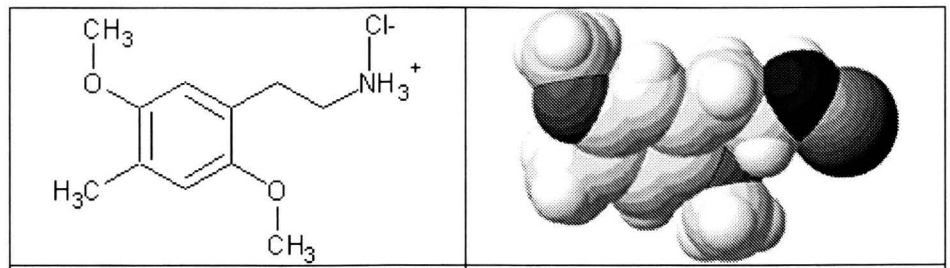
Procedure:

Personnel notes for procedure B: CAT

Into a suitable flask or beaker, place 10 grams of ephedrine (freebase), and then add in 75 milliliters of diethyl ether, and then place this flask or beaker into an ice bath, and chill to 0 Celsius. Thereafter, bubble into the ether mixture, 5 grams of dry hydrogen chloride gas. After the addition of the hydrogen chloride, continue to stir the ether mixture for about 30 minutes. Thereafter, place the entire mixture into a distillation apparatus, and distill-off the ether until only 80% of the total volume has been removed. When this happens, stop the distillation process, and recover the left over ether concentrate (after it has cooled to room temperature). Then filter-off the precipitated crystals of ephedrine hydrochloride, and then vacuum dry or air-dry these crystals. Then place 600 milliliters of methylene chloride into a clean beaker or flask, and then add in 7.2 grams of the ephedrine hydrochloride just prepared, and thereafter, add in 45 milliliters of glacial acetic acid, and then 300 milliliters of water. Then stir the entire two-phase mixture for about 10 minutes. Now, prepare a potassium permanganate solution by adding and dissolving 6 grams of potassium permanganate into 75 milliliters of water (a dark purple solution will result), and then slowly add this potassium permanganate solution to the ephedrine hydrochloride two-phase mixture over a period of about 10 to 15 minutes. During the addition, rapidly stir the two-phase reaction mixture, and maintain its temperature below 40 Celsius. After the addition of the potassium permanganate solution, moderately stir the two-phase reaction mixture at room temperature for about 90 minutes. After 90 minutes, add in 15 grams of sodium bisulfite, and then stir the entire two-phase reaction mixture for about 10 minutes. Thereafter, filter the entire two-phase reaction mixture to filter-off any insoluble materials, and then add to this filtered two-phase reaction mixture a sodium hydroxide solution prepared by adding and dissolving 60 grams (excess) of sodium hydroxide into 200 milliliters of water. Note: sodium hydroxide generates much heat when dissolved in water, so allow the solution to cool before using. After the addition of the sodium hydroxide solution, rapidly stir the two-phase reaction mixture for about 1 hour at room temperature. Thereafter, place the entire two-phase reaction mixture into a large seperatory funnel, and remove the lower methylene chloride layer, or recover it by decanting the upper aqueous layer. Then place the methylene chloride layer into a suitable sized beaker, and then add in 300 grams of a 5% sulfuric acid solution, and then rapidly stir the entire mixture for about 1 hour at room temperature. Now, place the entire two-phase mixture into a large clean seperatory funnel, and remove the lower aqueous layer, or recover the lower aqueous layer by decanting-off the upper methylene chloride mixture. Note: the methylene chloride can now be recycled by distillation. To the lower aqueous acidic layer, add in a sodium hydroxide solution prepared by adding and dissolving 30 grams (excess) of sodium hydroxide into 190 milliliters of water. Note: sodium hydroxide generates much heat when dissolved in water, so allow the solution to cool to room temperature before using. After the addition of the sodium hydroxide, extract the entire mixture, with three 100-millilter portions of methylene chloride, and after the extraction process, combine all methylene chloride portions, if not already done so, and then dry this combined methylene chloride portion by adding to it, 25 grams of anhydrous magnesium sulfate. Then stir the entire mixture for about 10 minutes, and thereafter, filter-off the magnesium sulfate. Then place the filtered methylene chloride layer into a distillation apparatus, and distill-off the methylene chloride at 40 Celsius until no more methylene chloride distills over. When this is the case, stop the distillation process, and then recover the left over remaining residue (after it has cooled). Finally, dissolve this residue into 100 milliliters of hexane, and then place this hexane mixture into an ice bath, and chill to 0 Celsius. Thereafter, bubble into this hexane mixture, 20 grams (excess) of dry hydrogen chloride gas. After the addition of the hydrogen chloride gas, stir the hexane mixture for about 1 hour at 0 Celsius, and then filter-off the precipitated CAT product. The filtered-off crystals can then be vacuum dried or air-dried.

Note: Other salts of the freebase CAT such as the sulfate, tartrate, citrate, and phosphate can be prepared by adding the corresponding acid to the hexane mixture of the freebase CAT compound obtained at the end of the above process. For sulfuric acid or tartaric acid, 1 mole of 98% sulfuric acid or d-tartaric acid should be added to 2 moles of freebase CAT in the hexane mixture. For citric acid or phosphoric acid, 1 mole of citric acid or phosphoric acid should be added to 3 moles of the freebase CAT in the hexane mixture. The hexane mixture after treatment with the corresponding acid in each of these cases is then evaporated using a distillation apparatus, or rotary evaporator to the point where only 80% of the total volume of the hexane mixture has been reduced. The resulting solvent concentrate can then be filtered to recover the product, which can then be vacuum dried or air-dried. All the salts of CAT are powerful stimulants/psychedelics, and the citrate salt may be more potent then the others. Note: if desired, the freebase CAT can be obtained by placing the freebase CAT in the hexane mixture, obtained at the end of the above process, into a distillation apparatus and removing the hexane until no more solvent is collected, and then recovering the left over residue of CAT (after it has cooled). For purified freebase CAT, the regular CAT should be used, and treated with a 25% sodium carbonate (or sodium hydroxide) solution with stirring to liberate the freebase CAT, which can then be extracted with methylene chloride (as in the procedure), and the solvent then removed to leave behind the purified freebase CAT.

0017. LE-25. 2C-D. *2-(2,5-dimethoxy-4-methylphenyl)ethanamine hydrochloride*



Structure	3D model

LE-25 forms colorless to slightly colored crystals (depending on purity) with a melting point of 214 Celsius. LE-25 is an interesting compound with strange stimulant and psychedelic activity. The compound tends to produce irregular stimulation with the extra ordinary ability to concentrate, thought process, and the ability to memorize information; as a result, it has been classified as a "Smart pill". However, the intoxicating effects of the drug may over ride or interfere with any intelligent effects as dose and personnel may very in the scope of the effects. Most persons under the influence of this drug have reported the drug to produce the usual stimulation effects, but with more of a sedative like atmosphere—persons may feel stimulated, but are relaxed at the same time. This is probably the result of the drugs psychedelic side. Persons under the influence of this drug may experience a degree of stimulation, with images, colors, lighting, and other similar effects being enhanced and experienced simultaneously under parallel conditions with the stimulation effects—effects ranging from bursts of energy to happiness, and confidence. Exact effects, duration, and rate of onset cannot be accurately determined because they all vary from person to person. Most literature reports this drug to be more of a "booster" for other drugs; for example, when admixed with CAT, amphetamine, methamphetamine, and similar drugs the intoxification and duration can almost double without fear of unsafe consequences such as overdose, or other medical complications.

Note: This substance is a controlled substance (psychedelic amphetamine) as listed in the US code of Federal regulations.

Toxicity: Low	**Rate of onset (average):** Moderate
Stimulation dosage (ingestion): 20 to 75 milligrams	**Duration of effects (average):** 4 to 6 hours (depending on the person)
Stimulation dosage (inhalation): 20 to 40 milligrams	**Habit forming potential:** Moderate
Stimulation dosage (injection): 10 milligrams +	**Estimated value U.S. (based on procedure):** $23 per gram

Procedure A: Preparation of LE-25

Materials:

1. 75 milliliters of 98% sulfuric acid	12. 17 grams of phosphorus oxychloride
2. 35 grams of p-nitrotoluene	13. 400 milliliters of dry hexane
3. 15 grams of aluminum turnings or aluminum foil pieces	14. 48 grams of nitromethane
4. 350 milliliters of diethyl ether	15. 1.7 grams of anhydrous ammonium acetate
5. 15 grams of anhydrous magnesium sulfate	16. 200 milliliters of 99% isopropyl alcohol
6. 100 milliliters of petroleum ether	17. 50 milliliters of toluene

7. 50 milliliters of a 25% sodium hydroxide solution	18. 11 grams of lithium aluminum hydride
8. 31.5 grams of dimethylsulfate	19. 535 milliliters of tetrahydrofuran (THF)
9. 150 milliliters of methylene chloride	20. 40 milliliters of 99% isopropyl alcohol
10. 10 grams of anhydrous magnesium sulfate	21. 12 milliliters of a 15% sodium hydroxide solution
11. 15.5 grams of N-methylformanilide	22. 10 grams of dry hydrogen chloride gas

Summary: LE-25 is prepared in a five-step process starting with the formation of toluhydroquinone. This toluhydroquinone is prepared by reaction of nitrotoluene with aluminum in the presence of sulfuric acid. After the reduction, the reaction mixture is extracted with ether, and the ether is then evaporated to recover a residue. This residue is then recrystallized from petroleum ether to recover the purified crystals of toluhydroquinone. The toluhydroquinone is then converted into 2,5-dimethoxytoluene by reaction with sodium hydroxide and dimethylsulfate. The reaction mixture is then extracted with methylene chloride, and the resulting methylene chloride mixture is then removed by evaporation. The resulting 2,5-dimethoxytoluene is then converted into 2,5-dimethoxy-4-methylbenzaldehyde by reaction with phosphorus oxychloride in the presence of N-methylformanilide. The reaction is then stirred overnight, and then filtered to recover the precipitated impure product, which is then extracted with hexane. The product is then collected after precipitation from the hot hexane, and the resulting 2,5-dimethoxy-4-methylbenzaldehyde is then converted into 2,5-dimethoxy-methyl-beta-nitrostyrene by condensation with nitromethane in the presence of ammonium acetate as catalyst. The resulting reaction mixture is then striped of excess nitromethane, and the resulting residue is then recrystallized from alcohol. The recovered 2,5-dimethoxy-methyl-beta-nitrostyrene is then purified by recrystallization from a toluene/hexane mixture. The purified 2,5-dimethoxy-methyl-beta-nitrostyrene is then finally converted into the desired LE-25 by reaction with lithium aluminum hydride in the presence of tetrahydrofuran as solvent. The reaction is rather simple, and thereafter, the desired product is recovered by treatment with dilute sodium hydroxide solution, followed by evaporation of the solvent. The remaining residue is then dissolved in alcohol, and the desired LE-25 then precipitated by hydrogen chloride. The hydrochloride salt is then purified by mixing it with ether.

Hazards: Wear gloves and use proper ventilation when handling p-nitrotoluene, which is a highly toxic substance. Avoid inhalation, and skin contact. Wear gloves when also handling concentrated sulfuric acid, sodium hydroxide solutions, phosphorus oxychloride, and hydrogen chloride, all of which are capable of forming skin burns. Wear gloves and avoid inhalation and skin contact when handling dimethyl sulfate and nitromethane, the previous one being highly toxic. Lithium aluminum hydride reacts violently with water, so store in airtight amber glass bottles away from moisture at all times. Nitromethane, diethyl ether, hexane, and tetrahydrofuran (THF) are highly flammable, and capable of forming explosive mixtures with air, so extinguish all flames before using.

Procedure:

Personnel notes for procedure A: LE-25

Step 1: Preparation of toluhydroquinone

Into a suitable flask or beaker (equipped with motorized stirrer or magnetic stirrer, and thermometer), place 1000 milliliters of water, followed by carefully adding 75 milliliters of 98% sulfuric acid. Note: sulfuric acid generates excessive heat when mixed with water, so use caution. Thereafter, slowly add in 35 grams of p-nitrotoluene. Then raise the temperature of the reaction mixture to 95 Celsius, if it has not already reached said temperature after addition of the sulfuric acid, and then slowly add in, in small portions, 15 grams of aluminum turnings (or aluminum foil pieces), over a period 4 hours, while rapidly stiring the reaction mixture and maintaining its temperature at 95 celsius. After the addition of the aluminum, continue to heat the reaction mixture at 95 Celsius for 30 minutes with rapid stirring. Thereafter, remove the heat source, and allow the reaction mixture to cool to room temperature. Then extract the entire reaction mixture with three 75-millilter portions of diethyl ether, and after the extraction process, combine all ether extracts (if not already done so), and then wash this combined ether portion with three 100-millilter portions of cold water. Note: during the extraction process and washing process, the ether will be the upper layer each time. After the washing process, dry the combined ether portion by adding to it, 15 grams of anhydrous magnesium sulfate, and then stir the entire mixture for about 10 minute—then filter-off the magnesium sulfate. Then place the filtered ether portion into a distillation apparatus, and distill-off the ether at 40 Celsius. When no more ether distills over or is collected, remove the left over remaining residue (after it has cooled) and then dissolve it into 100 milliliters of petroleum ether (more or less may be needed, so try out different volumes on your own). Finally, recrystallize the desired toluhydroquinone from this petroleum ether mixture. After the recrystallization process, vacuum dry or air-dry the collected crystals.

Step 2: Preparation of 2,5-dimethoxytoluene

Into a suitable beaker or flask (equipped with motorized stirrer or magnetic stirrer, and thermometer), place 250 milliliters of water, followed by 15.5 grams of toluhydroquinone (prepared in step1), followed by 40 milliliters of a 25% sodium hydroxide solution, followed by 31.5 grams of dimethylsulfate. During all the additions, rapidly stir the reaction mixture and maintain the temperature below 40 Celsius. The time needed for each addition should take no longer then 10 or 15 minutes. After the additions, continue to stir the reaction mixture at a temperature below 40 Celsius for 4 hours. After 4 hours, add in an additional 10 milliliters of a 25% sodium hydroxide solution, and then stir the reaction at room temperature for 2 days. After 2 days, add to the reaction mixture, 625 milliliters of water, and then stir the entire reaction mixture for 30 minutes. Then extract the entire reaction mixture with three 50-milliliter portions of methylene chloride, and after the extraction process, combine all methylene chloride portions (if not already done so), and then dry this combined methylene chloride portion by adding to it, 10 grams of anhydrous magnesium sulfate. Then stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Finally, place the filtered methylene chloride portion into a distillation apparatus, and remove the methylene chloride at 40 Celsius. When no more methylene chloride passes over or is collected, remove the left over remaining oily residue (after it has cooled), and then set aside for step 3. Note: this oily amber residue can be purified by vacuum distillation at 70 Celsius under a vacuum of 0.50 millimeters of mercury, but this is not necessarily needed for step 3.

Step 3: Preparation of 2,5-dimethoxy-4-methylbenzaldehyde

Into a suitable 3-neck flask equipped with a motorized stirrer, thermometer, and reflux condenser, place 17 grams of phosphorus oxychloride, followed by 15.5 grams of N-methylformanilide, and then heat this mixture at 100 Celsius for about 10 minutes. During this heating period, rapidly stir the reaction mixture. After heating for 10 minutes, add to the reaction mixture (through the top of the reflux condenser), 15 grams of 2,5-dimethoxytoluene (prepared in step 2), and after the addition (which should take no longer then a few minutes), continue to heat the reaction mixture at 100 Celsius for about 90 minutes. During this heating period, rapidly stir the reaction mixture. Thereafter, remove the heat source, and allow the reaction mixture to cool to room temperature. Then place the entire reaction mixture into a suitable sized beaker, and then add in 300 milliliters of warm water, and then stir the entire diluted reaction mixture overnight. The following day, filter-off any insoluble materials, and then vacuum dry or air-dry these filtered-off materials until they are as dry as possible. Then place the ugly filtered-off residue into a reflux apparatus, and then add in 75 milliliters of dry hexane, and then reflux the mixture at 70 Celsius for about 30 minutes. After 30 minutes, remove the heat source, and then decant-off or pour-off the hexane mixture from any insoluble materials (recover the hexane before it cools below 60 Celsius), and then place this poured-off hexane into a beaker, and keep for later. Note: keep any insoluble materials in the same reflux apparatus. Then add to this same reflux apparatus, 75 milliliters of more dry hexane, and then repeat the reflux at 70 Celsius for 30 minutes. Afterwards, remove the heat source, and then decant-off or pour-off the hexane (recover the hexane before it cools below 60 Celsius), and then add this hexane portion to the previous hexane portion. Note: when pouring off the hexane, leave any insoluble materials in the same reflux apparatus. Now, one last time, place 75 milliliters of more dry hexane into the same reflux apparatus, and repeat the reflux process at 70 celsius for 30 minutes. Afterwards, remove the heat source, and decant-off or pour-off the hexane (before it cools below 60 Celsius). Note: when removing the hexane this time, any left over insoluble residue can be discarded. Then add this last hexane portion to the previous two portions. By this time, some crystalline product will have precipitated from the previous hexane portions (as the hexane cools, crystals of the desired product begin to crystallize out). Allow the entire combined hexane portion to stand for several hours at room temperature, and afterwards, place the hexane mixture into an ice bath, and chill to 0 Celsius. Allow the combined hexane portions to sit at 0 Celsius for about 1 hour. Afterwards, filter-off the precipitated product, and then vacuum dry or air-dry the filtered-off product. Finally, recrystallize this dried product from 75 milliliters of boiling hexane. After the recrystallization process, filter-off any collected crystals, and then vacuum dry or air-dry the crystals. After thorough drying of the crystals, the result will be about 4 grams of the desired 2,5-dimethoxy-4-methylbenzaldehyde with a melting point of 84 Celsius.

Step 4: Preparation of 2,5-dimethoxy-4-methyl-beta-nitrostyrene

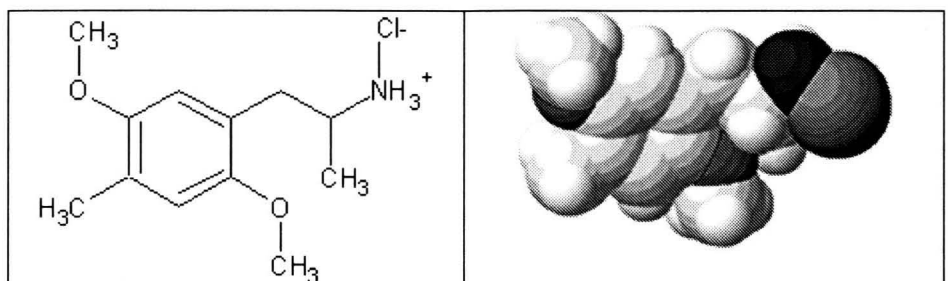
Into a suitable reflux apparatus, place 13.8 grams of 2,5-dimethoxy-4-methylbenzaldehyde (prepared in step 3), followed by 48 grams of nitromethane, followed by 1.7 grams of anhydrous ammonium acetate. Immediately thereafter, reflux the entire mixture at 100 Celsius for about 80 minutes. After refluxing for 80 minutes, quickly replace the reflux condenser with a standard condenser fitted with a receiver flask, and then distil-off the excess nitromethane at 102 Celsius. When no more nitromethane passes over or is collected, stop the distillation process, and then remove the left over remaining residue (after it has cooled to room temperature), and then place it into a suitable sized beaker. Then recrystallize this recovered residue from 160 milliliters of 99% isopropyl alcohol, and after the recrystallization process, vacuum dry or air-dry the filtered-off crystals. These dried crystals can then be recrystallized from 150 milliliters of a toluene/hexane solvent mixture prepared by mixing 50 milliliters of toluene with 100 milliliters of hexane. After the recrystallization process, vacuum dry or air-dry the filtered-off crystals, and then set aside for step 5.

Step 5: Preparation of LE-25

Into a 3-neck flask fitted with motorized stirrer, thermometer, reflux condenser, and addition funnel, place 11 grams of lithium aluminum hydride, followed by 480 milliliters of tetrahydrofuran. Then prepare a solution by adding and dissolving 13 grams of 2,5-dimethoxy-4-methyl-beta-nitrostyrene (prepared in step 4), into 55 milliliters of tetrahydrofuran. Then place this solution into the addition funnel, and then gently heat the tetrahydrofuran/lithium aluminum hydride mixture to about 30 Celsius, and then slowly add drop-wise, the 2,5-dimethoxy-4-methyl-beta-nitrostyrene/tetrahydrofuran solution over a period of about 55 minutes. Note: maintain the temperature below 40 Celsius during the addition, and rapidly stir the reaction mixture. After the addition, raise the temperature of the reaction mixture to 67 Celsius, and reflux the reaction mixture at this temperature for about 24 hours. After heating for 24 hours, remove the heat source, and allow the reaction mixture to cool to room temperature. Thereafter, place the reaction mixture into a suitable sized beaker, and then add in 40 milliliters of 99% isopropyl alcohol to destroy any unreacted lithium aluminum hydride. After the addition of the isopropyl alcohol, stir the entire reaction mixture for about 30 minutes. After 30 minutes, add in 12 milliliters of a 15% sodium hydroxide solution, followed immediately by 30 milliliters of water. Then stir the entire reaction mixture for about 1 hour. After 1 hour, filter the reaction mixture to remove insoluble impurities, and then wash the filtered-off impurities with two 80-milliliter portions of fresh tetrahydrofuran. Thereafter, combine both tetrahydrofuran washing portions, if not already done so, and then add this combined tetrahydrofuran portion to the filtered reaction mixture. Then place the reaction mixture into a distillation apparatus or rotary evaporator, and remove the tetrahydrofuran and isopropyl alcohol by distillation at 78 Celsius. When no more tetrahydrofuran or isopropyl alcohol passes over or is collected, stop the distillation process, and then recover the left over remaining residue (after it has cooled to room temperature). Then place this recovered left over residue into a clean beaker, and then add in 40 milliliters of 99% isopropyl alcohol. Then stir the entire mixture for about 30 minutes, and thereafter filter-off any insoluble impurities. Then place this filtered isopropyl alcohol mixture into an ice bath, and chill to 0 Celsius. Then bubble into this chilled isopropyl alcohol mixture, 10 grams of dry hydrogen chloride gas (excess), and after the addition, stir the entire mixture for about 1 hour at 0 Celsius. Then filter-off the precipitated crystals, and then vacuum dry or air-dry the crystals. Finally, purify the crystals by mixing them with 125 milliliters of diethyl ether, and then briefly stir the entire mixture for about 30 minutes. After this mixing process, filter-off the precipitated crystals, and then vacuum dry or air-dry them.

Note: Other salts of the freebase LE-25 such as the sulfate, tartrate, citrate, and phosphate can be prepared by adding the corresponding acid to the isopropyl alcohol mixture of the freebase LE-25 compound obtained at the end of step 5. For sulfuric acid or tartaric acid, 1 mole of 98% sulfuric acid or d-tartaric acid should be added to 2 moles of the freebase LE-25 in the isopropyl alcohol mixture. For citric acid or phosphoric acid, 1 mole of citric acid or phosphoric acid should be added to 3 moles of the freebase LE-25 in the isopropyl alcohol mixture. The isopropyl alcohol mixture after treatment with the corresponding acid in each of these cases is then filtered to recover the precipitated crystals. The crystals should then be vacuum dried or air-dried, and then mixed with ether (about 1 part salt to 12 parts ether), thereafter, the crystals can then be filtered-off, and then vacuum dried or air-dried. All the salts of LE-25 are powerful stimulants/psychedelics. Note: if desired, the freebase LE-25 can be obtained by placing the freebase LE-25 in the isopropyl alcohol mixture, obtained at the end of step 5, into a distillation apparatus and removing the isopropyl alcohol until no more solvent is collected, and then recovering the left over residue of LE-25 (after it has cooled). For purified freebase LE-25, the regular LE-25 salt should be used, and treated with a 25% sodium carbonate (or sodium hydroxide) solution with stirring to liberate the freebase LE-25, which can then be extracted with diethyl ether, and the solvent then removed to leave behind the purified freebase LE-25.

0018. DOM. STP. 2,5-dimethoxy-4-methylamphetamine hydrochloride. *1-(2,5-dimethoxy-4-methylphenyl)propan-2-amine*



Structure	3D model

DOM forms colorless to shinny white crystals with a melting point of 191 Celsius. The crystals are soluble in water, but insoluble in ether, methylene chloride, and hexane. The freebase is soluble in ether, methylene chloride and hexane. DOM is a very interesting and unique chemical compound that poses hallucinogenic activity related to the infamous LSD, but with added stimulation effects. DOM is capable of producing a unique blend of effects ranging from happiness, motivation, mental

awareness, focus, and energy bursts, leading up to extra ordinary heightening of sensations including sight, sound, smell, and touch. DOM is also capable of affecting the emotions whereby users may find themselves flip-flopping from one emotional mood to another during the coarse of intoxification—the primary emotions or moods, usually involve anger, love, and happiness—secondary moods may include hysterical out bursts and laughing, feelings of stupor, and/or sillyness. Users of this drug have stated that regardless of any hallucinogenic or stimulation effects, all throughout the intoxification, the drug produces a consistent "good feeling" without nausea or distress.

Note: This substance is a controlled substance (psychedelic amphetamine) as listed in the US code of Federal regulations.

Toxicity: High	**Rate of onset (average):** Slow (may take up to 2 hours for effects to be realized
Stimulation dosage (ingestion): 3 to 9 milligrams	**Duration of effects (average):** 14 to 20 hours (depending on the person)
Stimulation dosage (inhalation): unknown	**Habit forming potential:** Low
Stimulation dosage (injection): 0 (danger of overdose)	**Estimated value U.S. (based on procedure):** $23 per gram NOTE: based on street value of a $5 per 9 milligram hit = $555 per gram)

Procedure A: Preparation of DOM

Materials:

1. 210 milliliters of glacial acetic acid	8. 112 grams of potassium sodium tartrate
2. 13.7 grams of 2,5-dimethoxy-4-methylbenzaldehyde (see step 1 to 3 of procedure 0017.LE-25)	9. 25 grams of sodium hydroxide
3. 5 grams of anhydrous ammonium acetate	10. 225 milliliters of methylene chloride
4. 7.6 grams of nitroethane	11. 15 grams of anhydrous sodium sulfate
5. 4.7 grams of lithium aluminum hydride	12. 100 milliliters of hexane
6. 530 milliliters of diethyl ether	13. 10 grams of dry hydrogen chloride gas
7. 375 milliliters of a 8% sulfuric acid solution	

Summary: DOM is prepared in a familiar two-step process starting with the formation of 1-(2,5-dimethoxy-4-methylphenyl)-2-nitropropene. This intermediate is prepared by condensing 2,5-dimethoxy-4-methylbenzaldehyde with nitroethane in the presence of glacial acetic acid and ammonium acetate as catalyst. The reaction mixture is cooled, and the desired product precipitates after standing. The resulting 1-(2,5-dimethoxy-4-methylphenyl)-2-nitropropene is then converted into DOM by reaction with lithium aluminum hydride in the presence of diethyl ether. The reaction mixture is then refluxed, and then treated with dilute acid to destroy any salts. Thereafter, the reaction mixture is separated into two-phases, and the aqueous phase is then treated with potassium sodium tartrate. The resulting neutral mixture is then basified by the addition of sodium hydroxide solution. The alkaline mixture is then extracted with methylene chloride, and after removal of the solvent, the left over crystals are then recrystallized from hexane. The purified freebase compound is then dissolved into ether, and the corresponding DOM is then precipitated by the addition of hydrogen chloride.

Hazards: Extinguish all flames before using diethyl ether, nitroethane, or hexane, as they are all capable of producing explosive mixtures with air, and they are highly volatile—use proper ventilation. Wear gloves when handling lithium aluminum hydride, which is capable of causing skin irritation. Proper hand protection should also be exercised when handling sodium hydroxide, or glacial acetic acid.

Procedure:

Personnel notes for procedure A: DOM

Step 1: Preparation of 1-(2,5-dimethoxy-4-methylphenyl)-2-nitropropene

Into a suitable reflux apparatus, place 60 milliliters of glacial acetic acid, followed by 13.7 grams of 2,5-dimethoxy-4-methylbenzaldehyde (see steps 1 to 3 of procedure 0017.LE-25), and then stir the entire mixture to form a uniform mixture. Thereafter, add in 5 grams of anhydrous ammonium acetate, followed by 7.6 grams of nitroethane. Note: both additions should

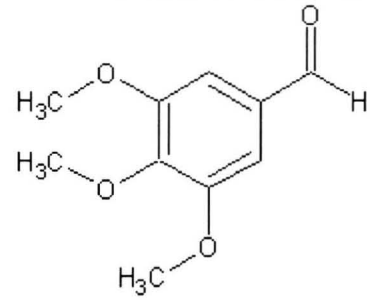
take no longer then 5 to 10 minutes. After both additions, reflux the entire mixture at 100 Celsius for about 60 minutes. After refluxing for about 60 minutes, remove the heat source, and allow the reaction mixture to cool to room temperature, and thereafter, dilute the reaction mixture by adding to it, an equal volume of ice water, and then place the diluted reaction mixture into an ice bath, and chill to 0 Celsius. Then let this diluted reaction mixture sit at 0 Celsius for about 1 hour. Thereafter, filter-off the precipitated crystals, and then vacuum dry or air-dry these collected crystals. Then recrystallize these dried crystals from 150 milliliters of boiling glacial acetic acid, and after the recrystallization process, vacuum dry or air-dry the crystals. The result will be about 7 grams of the desired 1-(2,5-dimethoxy-4-methylphenyl)-2-nitropropene with a melting point of 88 Celsius.

Step 2: Preparation of DOM

Into a suitable 3-neck flask equipped with motorized stirrer, thermometer, reflux condenser, and addition funnel, place 4.7 grams of lithium aluminum hydride followed by 190 milliliters of diethyl ether. Thereafter, briefly stir the mixture to dissolve the lithium aluminum hydride. Thereafter, prepare a solution by adding and dissolving 4.7 grams of 1-(2,5-dimethoxy-4-methylphenyl)-2-nitropropene (obtained in step 1) into 190 milliliters of diethyl ether, and then place this solution into the addition funnel. Then gradually add this solution in the addition funnel to the contents of the flask over a period sufficient to keep the reaction mixture below 40 Celsius at all times. During the addition, rapidly stir the reaction mixture. After the addition, reflux the entire reaction mixture at 50 to 60 Celsius for 2 hours with constant stirring. Thereafter, remove the heat source, and allow the reaction mixture to cool to room temperature. Then continue to stir the reaction mixture at room temperature over night. The next day, slowly add in 375 milliliters of an 8% sulfuric acid solution (to destroy lithium and aluminums salts), and stir the reaction mixture during the addition. After the addition, continue to stir the reaction mixture for about 1 hour. After 1 hour, place the entire reaction mixture into a seperatory funnel, and then remove the lower aqueous layer (the upper diethyl ether layer can be recycled or discarded if desired), and then wash this lower aqueous layer with one 75-milliliter portion of diethyl ether. After the washing portion, the aqueous layer will be the bottom layer (the upper ether layer can be recycled or discarded if desired). Now, to the recovered lower aqueous layer, add in 112 grams of potassium sodium tartrate, and then stir the entire aqueous mixture for about 1 hour at room temperature. Thereafter, add in a sodium hydroxide solution prepared by adding and dissolving 25 grams of sodium hydroxide (excess) into 150 milliliters of cold water. Note: sodium hydroxide generates much heat when dissolved in water, so allow the solution to cool before using. After the addition of the sodium hydroxide solution, continue to stir the reaction mixture for about 1 hour. After 1 hour, extract this entire aqueous alkaline mixture with three 75-milliliter portions of methylene chloride, and after the extraction process, combine all methylene chloride portions (if not already done so), and then quickly dry this combined methylene chloride portion by adding to it, 15 grams of anhydrous sodium sulfate. Thereafter stir the entire mixture for about 10 minutes, and then filter-off the sodium sulfate. Then place the filtered methylene chloride portions into a distillation apparatus, and remove the methylene chloride by distillation at 40 Celsius. When no more methylene chloride passes over or is collected, remove the left over remaining residue (after it has cooled), and then recrystallize this left over residue from 100 milliliters of hexane. After the recrystallization process, vacuum dry or air-dry the filtered-off crystals. Finally, dissolve the recovered crystals into 75 milliliters of diethyl ether, and then place this ether mixture into an ice bath, and chill to 0 Celsius, Then bubble into the ether mixture, 10 grams of dry hydrogen chloride gas (excess). After the addition of the hydrogen chloride gas, let the ether mixture stand at 0 Celsius for about 1 hour, and thereafter, filter-off the precipitated product. Then vacuum dry or air-dry the filtered-off crystals. The result will be about 4 grams of DOM with a melting point of 191 Celsius.

Note: Other salts of the freebase DOM such as the sulfate, tartrate, citrate, and phosphate can be prepared by adding the corresponding acid to the ether mixture of the freebase DOM compound obtained at the end of step 2. For sulfuric acid or tartaric acid, 1 mole of 98% sulfuric acid or d-tartaric acid should be added to 2 moles of the freebase DOM in the ether mixture. For citric acid or phosphoric acid, 1 mole of citric acid or phosphoric acid should be added to 3 moles of the freebase DOM in the ether mixture. The ether mixture after treatment with the corresponding acid in each of these cases is then filtered to recover the precipitated crystals. The crystals should then be vacuum dried or air-dried. All the salts of DOM are powerful stimulants/hallucinogens. For purified freebase DOM, the regular DOM salt should be treated with a 25% sodium carbonate (or sodium hydroxide) solution with stirring to liberate the freebase DOM, which can then be extracted with diethyl ether, and the solvent then removed to leave behind the purified freebase DOM. Note: the sulfate salt has a melting point of 131 Celsius.

Intermediate-0019. 3,4,5-TMB. *3,4,5-Trimethoxybenzaldehyde.*



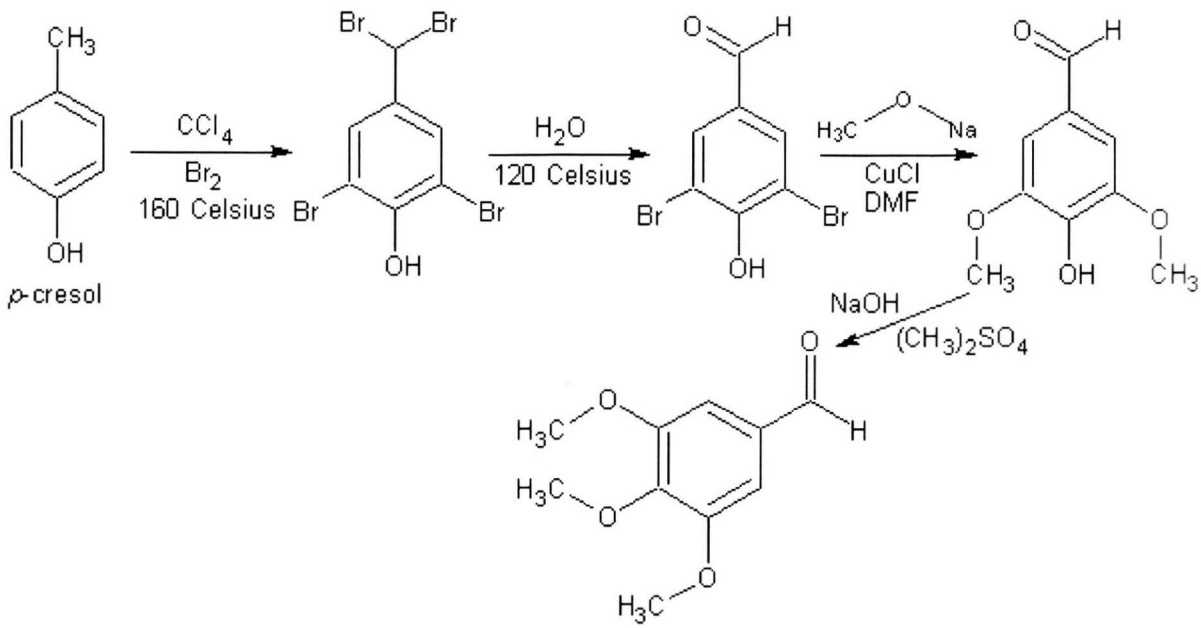
3,4,5-Trimethoxybenzaldehdye forms colorless to whitish crystals, which may be light brown or light yellow (depending on method of preparation), with a melting point of 75 Celsius. The crystals are widely used in a number of procedures for the preparation of psychedelic amphetamines—one of them being the famous mescaline.

Procedure A: Preparation of 3,4,5-Trimethoxybenzaldehyde from para-cresol

Materials:

1. 22 grams of para-cresol	6. 4 milliliters of dimethylformamide (DMF)
2. 300 milliliters of carbon tetrachloride	7. 2 grams of cuprous chloride
3. 130 grams of liquid bromine	8. 1 gram of powderd charcoal
4. 60 milliliters of 99% anhydrous methyl alcohol	9. 25 grams of dimethylsulfate
5. 21 grams of metallic sodium	10. 56 grams of a 30% sodium hydroxide solution

Summary: 3,4,5-trimethoxybenzaldehyde can be prepared in a two-step process starting with the formation of 3,4-dibromo-4-hydroxybenzaldehyde. This intermediate is readily prepared by treating para-cresol with bromine in the presence of carbon tetrachloride. After the initial reaction, water is added, and the resulting mixture is then refluxed. After chilling the reaction mixture, the desired product precipitates out, and is then easily collected by filtration. The resulting 3,4-dibromo-4-hydroxybenzaldehyde is then converted into the desired 3,4,5-trimethoxybenzaldehyde by reaction with sodium methoxide in the presence of cuprous chloride as catalyst under heat and pressure. Afterwards, the reaction mixture is then treated with dimethylsulfate and sodium hydroxide. The reaction mixture is then cooled, and the resulting product is then precipitated. It is then easily collected by filtration, washed, and then dried.



3,4,5-trimethoxybenzaldehyde

Hazards: Use caution when handling liquid bromine, which is highly toxic and corrosive, and capable of causing skin burns. Wear gloves when handling and use maximum ventilation. Avoid inhalation of methyl alcohol, which is poisonous. Wear gloves and use proper ventilation when handling dimethylsulfate, which is very toxic and can be absorbed through the skin. Wear gloves and use caution when handling metallic sodium, which reacts violently with water and various other chemicals.

Procedure:

Personnel notes for procedure A: 3,4,5-trimethoxybenzaldehyde

Step 1: Preparation of 3,4-dibromo-4-hydroxybenzaldehyde

Into a suitable 3-neck flask equipped with reflux condenser, thermometer, motorized stirrer, and addition funnel, place 22 grams of para-cresol, and 100 milliliters of carbon tetrachloride. Note: equip the top of the reflux condenser with a calcium chloride drying tube to keep moisture out. Thereafter, prepare a halogen mixture by adding and dissolving 65 grams of liquid bromine into 100 milliliters of carbon tetrachloride, and then place this solution into the addition funnel. Then cool the contents in the 3-neck flask to about 5 Celsius using an ice water bath, or other means, and thereafter, gradually add the bromine/carbon tetrachloride solution to the para-cresol mixture over a period of about 50 minutes. During the addition, continuously stir the reaction mixture, and maintain its temperature below 40 Celsius. After the addition of bromine/carbon tetrachloride solution, carefully heat the reaction mixture to about 150 Celsius (reflux), and thereafter, place another bromine solution into the same addition funnel. Prepare this bromine solution by adding and dissolving 65 grams of liquid bromine into 100 milliliter of carbon tetrachloride. Then gradually add this bromine/carbon tetrachloride solution to the reaction mixture over a period of about 90 minutes. During the addition, rapidly stir the reaction mixture, and maintain its temperature at about 150 Celsius. After the addition, continue to heat and stir the reaction mixture at 150 Celsius, for about 30 minutes. After 30 minutes, remove the heat source, and allow the reaction mixture to cool to room temperature. Thereafter, quickly add in 75 milliliters of water, and then reflux the entire reaction mixture for 2 hours with constant stirring. After 2 hours, remove the heat source, and allow the reaction mixture to cool to room temperature. Thereafter, pour the entire reaction mixture into a suitable sized beaker, and then chill this beaker in an ice bath. Note: use proper ventilation. When the temperature of the reaction mixture reaches about 0 Celsius, let the reaction mixture stand for 1 hour at 0 Celsius. Then filter-off the precipitated product, and then wash this filtered-off product with four 100-milliliter portions of cold water. Note: the filtered reaction mixture will be composed of two-phases, an upper aqueous layer, and a lower carbon tetrachloride layer—this lower layer can be recycled or discarded if desired. After the filtered-off product has been washed and dried, it will weigh about 45 grams, and have a melting point of about 182 Celsius.
Note: In most literature, all the carbon tetrachloride used in step 1, is substituted with the same volume of ortho-dichlorobenzene. However, in this step, carbon tetrachloride is used instead. If desired, the ortho-dichlorobenzene can be used in place of the carbon tetrachloride, and may result in improved yields.

Step 2: Preparation of 3,4,5-trimethoxybenzaldehyde

Into a suitable beaker, immersed in an ice bath, place 50 milliliters of 99% anhydrous methyl alcohol, and then allow this alcohol to chill to 0 Celsius. Thereafter, slowly add in, in small portions, 21 grams of metallic sodium. During the addition of the metallic sodium, rapidly stir the reaction mixture using a magnetic stirrer, or motorized stirrer. Also during the addition, maintain the reaction mixtures temperature below 40 Celsius. After the addition of the metallic sodium, add in 10 milliliters of additional 99% anhydrous methyl alcohol, and then stir the reaction mixture for 1 hour until it fully cools to room temperature. Then, place this methyl alcohol mixture into a single neck flask, and then add to it, 4 milliliters of dimethylformamide (DMF), followed by 2 grams of cuprous chloride, followed by 28 grams of the product obtained in step 1. Thereafter, place a suitable sized balloon over the flask, and secure the balloon to the flask using a metal ring clamp. Then heat the contents in the flask to about 120 Celsius for about 90 minutes. Note: the balloon will inflate and deflate sporadically during the heating process. The balloon is deigned to keep the contents of the flask under pressure to properly carryout the reaction. If during the heating process, the balloon pops or explodes, quickly replace with another one, and continue the operation for the necessary amount of remaining time. ***Note:*** *the pressure process just described, whereby a balloon is placed over a flask, can be substituted by using a conventional steel pipe with threads at both ends. To carryout the steel pipe technique, pour all necessary materials (as described for the balloon technique), into a large thick walled stainless steel pipe, and then seal both ends with the corresponding steel caps. Note: the steel pipe should be large enough so that after all ingredients have been added, only 1/3 or less of its total volume has been filled. The threads at each end should be wrapped with Teflon tape prior to screwing in the end caps. Then place the entire pipe, and submerge it into an oil bath and heat at the desired temperature for the desired time. Note: this process can be dangerous and can lead to pressure explosions. Carryout the process in an area that can contain any such explosion, and maintain a safe distance away during the operation—just to be on the safe side.* After the heating process, remove the heat source, and allow the reaction mixture to cool to room temperature. Thereafter, pour the entire reaction mixture into a distillation apparatus, and distil-off the methyl alcohol at 68 Celsius. When no more methyl alcohol passes over,

or is collected, allow the left over remaining residue to cool to room temperature, and thereafter, add to it (without removing it from the distillation apparatus), 100 milliliters of water, and then reflux this new mixture at 90 Celsius until most of the residue dissolves (simply replace the conventional condenser with a reflux condenser). After most of the residue dissolves, stop the reflux process, and then remove the aqueous mixture from the apparatus before it fully cools, and place it into a suitable sized beaker. Then allow the contents of this beaker to cool to room temperature, and thereafter, place the beaker into an ice bath, and chill to 0 celsius. Thereafter, let this mixture stand at 0 Celsius for about 2 hours. After 2 hours, filter-off the precipitated product, and then vacuum dry or air-dry this filtered-off solid (filter-cake). Then dissolve the dry filtered-off solid into 150 milliliters of water, and then add in 1 gram of powdered charcoal (regular charcoal, no fancy charcoal such a "quick lite"). Then heat this mixture for about 15 minutes at 90 Celsius. Afterwards, stop the heating process, and filter the mixture hot. Then allow the filtered mixture to cool to about 40 Celsius. Then place it into a clean 3-neck flask, fitted with a thermometer, reflux condenser, motorized stirrer, and two addition funnels, and then apply heat to the mixture to maintain the temperature at 40 Celsius. Then quickly place 25 grams of dimethylsulfate into one of the addition funnels, and then place 56 grams of a 30% sodium hydroxide solution into the second addition funnel. Thereafter, gradually add drop-wise, the dimethylsulfate (in addition funnel 1), and the sodium hydroxide solution, drop-wise (from addition funnel 2), both simultaneously and over a period of about 1 hour. Note: simply add both the dimethylsulfate and sodium hydroxide solution at a relatively similar pace. During both additions, rapidly stir the reaction mixture, and maintain its temperature at 40 Celsius. After both additions, raise the temperature of the reaction mixture to 50 Celsius, and then continue rapid stirring for about 15 minutes. Afterwards, remove the heat source, and allow the reaction mixture to cool to room temperature. Then place the reaction mixture into an ice bath, and chill to 0 Celsius. Then stir the reaction mixture at 0 Celsius for about 1 hour. Finally, filter-off the precipitated product, wash with three 100-milliliter portions of cold water, and then vacuum dry or air-dry the product. The result will be about 16 grams of the desired 3,4,5-trimethoxybenzaldehyde with a melting point of 75 Celsius.

Procedure B: Preparation of 3,4,5-Trimethoxybenzaldehyde from vanilla extract (food grade)

Materials:

1. 75 milliliters to 118 milliliters of grocery store brand vanilla extract	5. 23 milliliters of 35 to 38% hydrochloric acid
2. 300 milliliters of diethyl ether	6. 200 grams of a 22% sodium bisulfite solution
3. 10 grams of anhydrous magnesium sulfate	7. 54 grams of anhydrous sodium carbonate
4. 100 milliliters of methanol	8. 150 milliliters of methylene chloride
5. 17.6 grams of liquid bromine	9. 15 grams of anhydrous sodium sulfate
2. 50 milliliters of an ice cold 70% methanol solution	10. 85 milliliters of acetone
3. 12 grams of sodium hydroxide	11. 15 grams of dimethylsulfate
4. 100 milligrams of copper powder	12. 1.6 milliliters of a 10% potassium hydroxide solution in methanol

Summary: 3,4,5-trimethoxybenzaldehyde can be made in a 4-step process starting with the formation of 5-bromovanillin. 5-bromovanillin is readily prepared by reacting vanillin with liquid bromine at low temperature. Vanillin is readily obtained from regular store bought vanilla extract, by treatment with ether, followed by evaporation, and recrystallization from hot water. The 5-bromovanillin is converted into 5-hydroxyvanillin by reaction with sodium hydroxide in the presence of a small amount of copper powder. After the initial reaction, the mixture is treated with sodium bisulfite to precipitate the adduct salt, which is filtered-off, and then dissolved into a sodium carbonate solution to liberate the aldehyde. This compound is then extracted into methylene chloride, and recovered by evaporation. The corresponding 5-hydroxyvanillin is then converted into the desired 3,4,5-trimethoxybenzaldehyde by reaction with dimethylsulfate in the presence of acetone, sodium carbonate, and a small amount of a 10% potassium hydroxide solution in methanol. The reaction mixture is refluxed for a short period of time, and then cooled to ice bath temperatures. The chilled reaction mixture is then filtered to recover the desired 3,4,5-trimethoxybenzaldehyde.

Hazards: Use caution when handling liquid bromine, which is highly toxic and corrosive, and capable of causing skin burns. Wear gloves when handling and use maximum ventilation. Avoid inhalation of methyl alcohol (methanol), which is poisonous. Wear gloves and use proper ventilation when handling dimethylsulfate, which is very toxic and can be absorbed through the skin. Extinguish all flames before using diethyl ether, and acetone, both of which are capable of forming explosive mixtures with air. Wear gloves when handling concentrated hydrochloric acid, and sodium hydroxide as both are capable of causing skin burns, and irritation.

Procedure:

Personnel notes for procedure B: 3,4,5-trimethoxybenzaldehyde

Step 1: Extraction of vanillin from store bought vanilla extract

Pour a large bottle (75 milliliters to 118 milliliters) of grocery store brand vanilla extract into a suitable beaker, and then add in 50 milliliters of warm water. Then extract this entire mixture with three 50-milliliter portions of diethyl ether, and after the extraction process, combine all ether portions (if not already done), and then dry this combined ether portion by adding to it, 10 grams of anhydrous magnesium sulfate. Then stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Then place this filtered ether mixture into a distillation apparatus, and distill-off the ether at 40 Celsius. When no more ether passes over or is collected, stop the heating process, and recover the left over remaining residue (after it has cooled to room temperature), and then vacuum dry or air-dry this collected residue. Thereafter, set this dry residue aside just for a moment. Now, depending on how much residue you have (based on what quantity of grocery store vanilla extract you purchased), add your collected left over residue into heated water contained in suitable sized beaker. In other words, place 20 milliliters of water per 1 gram of your residue into a breaker, and heat to 80 Celsius—thereby, add in your residue. After you add in the residue, continue to heat the water mixture at 80 Celsius with moderate stirring for about 15 minutes, and then quickly filter this water mixture (before it cools), and then place the filtered water mixture into a clean beaker, and allow it to cool to room temperature—whereby crystals of vanillin will form. After the water mixture has cooled to room temperature, place it into an ice bath (or use a freezer), and allow the mixture to stand at 0 Celsius for 1 hour. Then filter-off the precipitated crystals of vanillin, and then vacuum dry or air-dry the crystals. Note: the crystals should be stored in airtight bottles in a cool place to prevent oxidation. Note: there are numerous modifications to this extraction process.

Step 2: Preparation of 5-bromovanillin

Into a suitable beaker or flask, equipped with thermometer, and motorized stirrer or magnetic stirrer, place 15 grams of vanillin, followed by 100 milliliters of methanol. Thereafter, briefly stir the mixture to dissolve all the vanillin. Then place this vanillin solution into an ice bath, and chill to 0 Celsius. Thereafter, slowly add drop-wise, 17.6 grams of liquid bromine over a period sufficient enough to keep the vanillin mixture below 20 Celsius. During the addition, rapidly stir the reaction mixture. After the addition, continue to stir the reaction mixture at a temperature below 20 Celsius for about 30 additional minutes. Thereafter, remove the ice bath, and then stir the reaction mixture at room temperature for about 30 minutes. Afterwards, re-put the reaction mixture into an ice bath, and then chill once again to 0 Celsius. When its temperature reaches 0 Celsius, add in 50 milliliters of water over a period of about 10 minutes. During the addition, moderately stir the reaction mixture, and after the addition, continue to stir the reaction mixture at a temperature around 0 Celsius for about 30 minutes. Finally, after stirring for 30 minutes, filter-off the precipitated product, wash with four 50-milliliter portions of cold water, followed by one portion of 50 milliliters of an ice cold 70% methanol solution. After the washings, vacuum dry or air-dry the crystals. The result will be 21 grams of bromovanillin as pale yellow crystals with a melting point of 164 Celsius.

Step 3: Preparation of 5-hydroxyvanillin

Into a suitable flask or beaker, place 150 milliliters of water, followed by 12 grams of sodium hydroxide. Thereafter, stir the mixture to dissolve all of the sodium hydroxide. Note: as usual, sodium hydroxide generates much heat when dissolved in water, so allow this solution to cool to about 40 Celsius before proceeding. Thereafter, add in 10 grams of 5-bromovanllin (prepared in step 2), followed by 100 milligrams of copper powder. During both additions, rapidly stir the reaction mixture and maintain the temperature below 45 Celsius. Then continue to stir the reaction mixture until a white precipitate can be seen forming. When this point happens, pour the entire reaction mixture into a reflux apparatus, fitted with water trap, and nitrogen purge adapter, and then begin a steady stream of nitrogen gas into the apparatus to flush out air. Thereafter, reflux the reaction mixture at about 100 Celsius for 6 hours with rapid stirring. After refluxing for 6 hours, stop the reflux process, and allow the reaction mixture to cool to room temperature. Then add to the cooled reaction mixture, 23 milliliters of 35 to 38% hydrochloric acid (muriatic acid of 31% can be used), and then stir the acidified reaction mixture for about 30 minutes. Afterwards, extract the entire reaction mixture with three 50-milliliter portions of diethyl ether, and after the extraction process, combine all ether extracts (if not already done so), and then thoroughly mix this combined ether portion with 200 grams of a 22% sodium bisulfite solution. After the addition of the sodium bisulfite solution, rapidly stir the entire mixture for about 30 minutes. Thereafter, filter-off the precipitated solids (which will be composed of a bisulfite addition adduct), and then wash this filtered-off solid with 50 milliliters of cold water, and then vacuum dry or air-dry it. Thereafter, add this dried filtered-off solid to 400 grams of a 10% sodium carbonate solution contained in a suitable beaker, and then stir the entire mixture for about 1 hour. Note: this 10% sodium carbonate solution can be prepared by adding and dissolving 40 grams of anhydrous sodium carbonate into 360 milliliters of water. Note: sodium carbonate generates heat when dissolved in water, and the decahydrate may precipitate out of solution so stir the entire alkaline mixture thoroughly to dissolve any precipitated solids, before using this

sodium carbonate solution. After the addition of the sodium carbonate solution, and after stirring the mixture for 1 hour, extract the entire mixture with three 50-milliliter portions of methylene chloride, and after the extraction process, combine all methylene chloride portions (if not already done so), and then dry this combined methylene chloride portion by adding to it, 15 grams of anhydrous sodium sulfate. Then stir the entire mixture for about 10 minutes, and then filter-off the sodium sulfate. Finally, place the filtered methylene chloride portion into a distillation apparatus, and distill-off the methylene chloride at 40 Celsius. When no more methylene chloride passes over or is collected, stop the heating, and then collect the left over remaining solids (after they have cooled). Then place these solids aside for step 4.

Step 4: Preparation of 3,4,5-timethoxybenzaldehyde

Into a suitable sized flask, fitted with thermometer, motorized stirrer or magnetic stirrer, and reflux condenser, place 8.3 grams of 5-hydroxyvanillin (prepared in step 3), followed by 85 milliliters of acetone, followed by 15 grams of dimethylsulfate, followed by 14 grams of anhydrous sodium carbonate, and then followed by 1.6 milliliters of a 10% potassium hydroxide solution in methanol. Thereafter, reflux this entire mixture for 6 hours at 60 Celsius with vigorous stirring. After refluxing for 6 hours, quickly replace the reflux condenser with a conventional condenser (fitted with receiver flask), and then distill-off the acetone and any other liquids by heating to 100 Celsius. When no more liquid (acetone, water, or methanol) passes over or is collected, stop the distillation process, and then recover the left over remaining solid residue (after it has cooled). Then place this left over residue into a suitable sized beaker, and then add to it, 70 milliliters of water, and then rapidly stir this mixture for about 30 minutes at room temperature. After stirring for about 30 minutes, place the mixture into an ice bath, and chill to 0 Celsius. Then continue rapid stirring at 0 Celsius for about 30 minutes. Thereafter, filter-off the light brownish crystals, and then wash these crystals with three 25-milliliter portions of ice cold water, and then vacuum dry or air-dry the crystals. The result will be about 9 grams of the desired 3,4,5-trimethoxybenzaldehdye with a melting point of about 75 Celsius.

Procedure C: Preparation of 3,4,5-Trimethoxybenzaldehyde from syringaldehyde

Materials:

1. 50 milliliters of 99% anhydrous methanol	7. 80 milliliters of ethyl acetate
2. 2.4 grams of metallic sodium	8. 15 grams of anhydrous magnesium sulfate
3. 8.9 grams of bromovanillin (see procedure B, intermediate-0019, step 2)	9. 8.4 grams of potassium carbonate
4. 1.5 grams of cuprous bromide	10. 5.1 grams of dimethylsulfate
5. 75 milliliters of dimethylformamide (DMF)	11. 75 milliliters of cyclohexane
6. 100 milliliters of a 10% hydrochloric acid solution	

Summary: 3,4,5-trimethoybenzaldehyde can be prepared directly from bromovanillin by first, reacting the bromovanillin with metallic sodium in the presence of cuprous bromide in methanol. The reaction mixture is then distilled simultaneously to recover the methanol. After the methanol and any water has been removed, the reaction mixture is extracted with ethyl acetate, and the resulting acetate mixture then evaporated to recover the intermediate, syringaldehyde. The syringaldehyde is then converted into the desired 3,4,5-trimethoxybenzaldehdye by reaction with dimethylsulfate in the presence of potassium carbonate and dimethylformamide. The reaction is generally mild, and after stirring for a short period of time, the reaction mixture is drowned in ice water, and the resulting precipitated product is then filtered-off, dried, and then purified by recrystallization from cyclohexane.

Hazards: Wear gloves when handling dimethylsulfate and use proper ventilation. Dimethylsulfate is toxic, and easily absorbed through the skin, avoid inhalation and skin contact. Extinguish all flames before using methanol, ethyl acetate, and cyclohexane, all of which are very flammable. Wear gloves when handling metallic sodium, and keep away from water and other chemicals.

Procedure:

Personnel notes for procedure C: 3,4,5-trimethoxybenzaldehyde

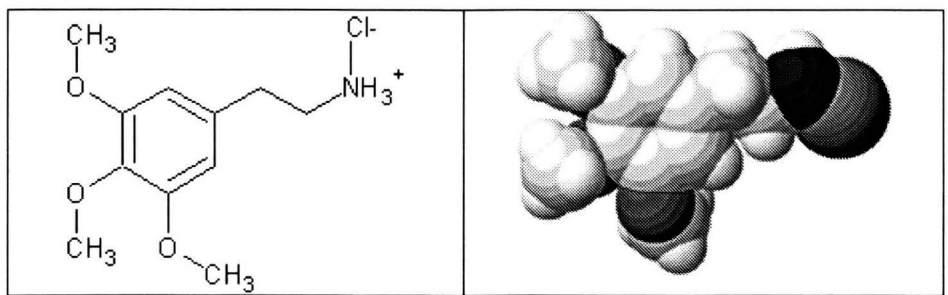
Step 1: Preparation of syringaldehyde (3,5-dimethoxy-4-hydroxybenzaldehyde

Into a suitable distillation apparatus equipped with thermometer and motorized stirrer, place 50 milliliters of 99% anhydrous methanol, and then place the flask into an ice water bath and chill to about 10 Celsius. Thereafter, slowly add, in small portions, 2.4 grams of metallic sodium over a period of time sufficient to keep the reaction mixture below 40 Celsius. After the addition of the metallic sodium, continue to stir the reaction mixture for about 30 minutes. Then, heat the reaction mixture to about 70 Celsius, and thereafter, add in a solution prepared by adding 8.9 grams of bromovanillin and 1.5 grams of cuprous bromide into 25 milliliters of dimethylformamide (DMF), all at once and in one portion. Note: during the addition, moderately stir the reaction mixture. After the addition, continue to heat and distill the reaction mixture at 70 Celsius with constant stirring for about 1 hour or until no more methanol and water distills over. Note: during the 1-hour or more heating and distilling process, slowly raise the temperature of the reaction mixture to 100 Celsius. When no more methanol and/or water distills over, stop the heating and distillation process, and allow the remaining left over contents of the reaction mixture to cool to temperature before collecting. Thereafter, place the collected left over contents into a suitable sized beaker, and then add in 100 milliliters of a 10% hydrochloric acid solution, and then quickly add in 50 grams of crushed ice. Then rapidly stir the mixture for 30 minutes. After stirring for 30 minutes, extract the entire mixture with two 40-milliliter portions of ethyl acetate, and after the extraction process, combine all ethyl acetate portions, if not already done so, and then wash the combined ethyl acetate portion with two 25-milliliter portions of ice cold water. Note: after each extraction and washing portion, the ethyl acetate will be the upper layer each time. After the washing process, dry the washed ethyl acetate portion by adding to it, 15 grams of anhydrous magnesium sulfate, and then stir the whole mixture for about 10 minutes—thereafter, filter-off the magnesium sulfate. Finally, place the filtered ethyl acetate portion into a distillation apparatus, and distill-off the ethyl acetate at 78 Celsius. When no more ethyl acetate passes over or is collected, stop the distillation process, and recover the left over remaining residue (after it has cooled). The result will be the desired syringaldehyde with a melting point of 108 Celsius.

Step 2: Preparation of 3,4,5-trimethoxybenzaldehyde

Into a suitable flask, equipped with thermometer, motorized stirrer or magnetic stirrer, place 6.6 grams of the product (obtained in step 1), followed by 8.4 grams of potassium carbonate, followed by 5.1 grams of dimethylsulfate, and finally followed by 50 milliliters of dimethylformamide (DMF). Thereafter, stir the reaction mixture for about 90 minutes without the aid of any cooling means; in other words, any heat that is generated by the reaction should be ignored. After stirring for 90 minutes, pour the entire reaction mixture into 500 milliliters of ice water (contained in a suitable beaker), and then stir the mixture until the ice melts. Afterwards, filter-off the precipitated product, wash with two 50-millilter portions of ice cold water, and then vacuum dry or air-dry the washed product. Finally, recrystallize the dried product from 75 milliliters of cyclohexane. After the recrystallization process, vacuum dry or air-dry the filtered-off product.

0020. MESCALINE. M-345. 3,4,5-trimethoxyphenethylamine hydrochloride. *2-(3,4,5-trimethoxyphenyl)ethanamine hydrochloride*



Structure	3D model

MESCALINE forms colorless to white glistening crystals with a melting point of 181 Celsius. The crystals are soluble in water, and alcohol, but insoluble in ether. MESCALINE is an interesting compound that exists naturally (as the freebase) in peyote (a small dumpling cactus—has small round shape with the appearance of tufts of soft fuzz instead of the usual spines or quills), that grows in the southwestern United States, and in northern Mexico—can be isolated from the flowering heads of *lophophora williamsii, cactaceae.* It has been used by native Indians for thousands of years in "vision" quests and spiritual rituals, where its hallucinogenic properties are clearly evident. MESCALINE is a powerful and potent psychedelic amphetamine that produces hallucinogenic and stimulant effects when ingested. These effects can last for up to 12 hours, with a strong sense of intoxification coupled with intense energy. The stimulation effects are similar to methamphetamine, and the hallucinogenic activity resembles other psychedelic compounds, but with a special twist not found in most other psychedelics. Along with the usual hallucinogenic effects, including enhancements of sight, sounds, smells, feelings, and touch, the drug tends to produce severe psychological responses in the brain. These psychological effects include what many have stated as "spiritual journeys", where users have been able to see themselves and analyze themselves to the point of deep emotional contact with ones self—kind of like self meditation, but multiplied by 100. Some have called MESCALINE a "spiritual" drug,

as have the native Indians, which is clearly understandable due to the severe emotional (meditative) states it produces. These meditative states, along with the usual other hallucinogenic effects and stimulant effects, makes MESCALINE a highly studied psychedelic amphetamine that is used as the standard for comparing all other hallucinogenic and psychedelic amphetamines. An interesting fact about MESCALINE, is that produces very few side-effects (that methamphetamine does) and it produces no withdrawal symptoms—however, chronic use may lead to withdrawal symptoms. Users of MESCALINE, after coming-off the drug, have stated a complete and relative state of satisfaction after experiencing such a trip. In other words, after coming off the drug, users will often feel very satisfied, relaxed, and generally good—without feelings of withdrawal or sickness. First time users of MESCALINE are likely to experience mild nausea after the first hour or so; however, the nausea is short lived. **Note: This substance is a controlled substance (psychedelic amphetamine) as listed in the US code of Federal regulations.**

Toxicity: Low	**Rate of onset (average):** Moderate (may take up to 1 hour for effects to be realized)
Stimulation dosage (ingestion): 200 to 400 milligrams	**Duration of effects (average):** 10 to 12 hours (depending on the person)
Stimulation dosage (inhalation): 150 to 250 milligrams	**Habit forming potential:** Very low
Stimulation dosage (injection): 50 to 100 milligrams	**Estimated value U.S. (based on procedure):** $20 per gram (estimated street value: $7 per 300 milligram hit = $23 per gram)

Procedure A: Preparation of MESCALINE

Materials:

1. 10 grams of 3,4,5-trimethoxybenzaldehyde (see intermediate-0019. 3,4,5-TMB)	8. 450 milliliters of a 10% sulfuric acid solution
2. 20 milliliters of nitromethane	9. 150 grams of potassium sodium tartrate
3. 10 milliliters of cyclohexylamine	10. 65 grams of sodium hydroxide
4. 300 milliliters of methanol	11. 450 milliliters of methylene chloride
5. or 500 milligrams of anhydrous ammonium acetate	12. 20 grams of anhydrous sodium sulfate
6. 750 milliliters of dry diethyl ether	13. 30 milliliters of 99% isopropyl alcohol
7. 6 grams of lithium aluminum hydride	14. 10 grams of dry hydrogen chloride gas

Summary: MESCALINE is prepared in a two-step process starting with the formation of beta-nitro-3,4,5-trimethoxystyrene. This intermediate is simply prepared by condensing 3,4,5-trimethoxybenzaldehyde with nitromethane in the presence of either glacial acetic acid and cyclohexylamine, or in the presence of ammonium acetate catalyst. The reaction mixture in either case, is refluxed, and then allowed to cool. The desired intermediate product is then collected by either precipitation, or solvent removal. The collected intermediate can then be purified by recrystallization from hot methanol. The purified intermediate, is then converted into the desired MESCALINE by reaction with lithium aluminum hydride in the presence of diethyl ether. After the reduction, the reaction mixture is gently refluxed for 2 days, and then acidified with dilute sulfuric acid. The reaction mixture is then separated into layers, and the aqueous layer is then basified by the addition of potassium sodium tartrate, followed by sodium hydroxide. The alkaline reaction mixture is then extracted with methylene chloride. The methylene chloride is then simply evaporated, and the left over residue is then dissolved into isopropyl alcohol, and the desired MESCALINE is then precipitated by the addition of hydrogen chloride gas. The crystals are then filtered-off, and then dried.

Hazards: Extinguish all flames before using diethyl ether, nitromethane, cyclohexylamine, and isopropyl alcohol, all which are very flammable. Diethyl ether and nitromethane are capable of producing explosive mixtures in air, so avoid any source of ignition when handling, and use proper ventilation. As usual, wear gloves when handling any acids or solutions thereof, as well as strong bases such as sodium hydroxide.

Procedure:

Personnel notes for procedure A: MESCALINE

Step 1:Preparation of beta-nitro-3,4,5-trimethoxystyrene (method 1)

Into a suitable reflux apparatus, place 10 grams of 3,4,5-trimethoxybenzaldehyde, followed by 20 milliliters of nitromethane, followed by 10 milliliters of cyclohexylamine, and then followed by 100 milliliters of glacial acetic acid. Thereafter, reflux the entire mixture at 100 Celsius for about 1 hour. After refluxing for 1 hour, remove the heat source, and allow the reaction mixture to cool to room temperature. Then pour the entire reaction mixture into a suitable sized beaker, and then slowly add to it, 200 milliliters of cold water. During the slow addition of the cold water, rapidly stir the reaction mixture. During and after the addition, a precipitate will form, and after the addition of the cold water, continue to stir the reaction mixture at room temperature for about 30 minutes. Thereafter, filter-off the precipitated product, wash with two 50-milliliter portions of cold water, and then vacuum dry or air-dry the precipitate. Finally, recrystallize this dried product from 150 milliliters of boiling methanol, and after the recrystallization process, vacuum dry or air-dry the filtered-off crystals. The result will be about 9 grams of the desired beta-nitro-3,4,5-trimethoxystyrene as bright yellow crystals.

Step 1: Preparation of beta-nitro-3,4,5-trimethoxystyrene (method 2)

Into a suitable reflux apparatus, place 10 grams of 3,4,5-trimethoxybenzaldehdye, followed by 20 milliliters of nitromethane, and then followed by 500 milligrams of anhydrous ammonium acetate. Then reflux the entire mixture at 100 Celsius for 2 hours. After refluxing for 2 hours, quickly replace the reflux condenser with a standard cold water condenser (fitted with a receiver flask), and then distill-off the excess nitromethane at 102 Celsius. When no more nitromethane passes over or is collected, stop the distillation process, and then recover the left over remaining oily residue (after it has cooled to room temperature), and then place this recovered oil into a clean beaker. Now, dissolve this oil into 150 milliliters of boiling methanol, and then stir the entire mixture for about 5 minutes, and before the methanol cools, quickly filter-off any insoluble impurities. Thereafter, allow the methanol solution to cool to room temperature—whereby crystals of the desired product will form. Thereafter, filter-off the crystals, and then vacuum dry or air-dry them. Them recrystallize these crystals from 150 milliliters of boiling methanol, and after the recrystallization process, vacuum dry or air-dry the filtered-off crystals.

Step 2: Preparation of MESCALINE

Into a suitable reflux apparatus (including a motorized stirrer), place 600 milliliters of dry diethyl ether, and then add in 6 grams of lithium aluminum hydride, and then gently reflux the mixture at about 40 Celsius. Note: attach a calcium chloride drying tube to the top of the condenser to keep moisture out. Then, add in small portions (through the top of the reflux condenser—briefly remove the calcium chloride drying tube for each addition, and then quickly replace it after each addition) 7.2 grams of the product obtained in step 1. The addition should take no longer the 10 to 15 minutes. During the addition, rapidly stir the reaction mixture. After the addition, continue to reflux the reaction mixture at 40 Celsius for about 48 hours with rapid stirring. After 48 hours, remove the heat source, and allow the reaction mixture to cool to room temperature. Then pour the entire reaction mixture into a clean dry beaker, and then slowly add to it, 450 milliliters of a 10% sulfuric acid solution. Note: during the addition of the sulfuric acid, rapidly stir the reaction mixture by means of a magnetic stirrer, or other means. After the addition of the sulfuric acid, stir the entire acidified mixture for about 30 minutes. After 30 minutes, place the entire two-phase mixture into a seperatory funnel, and remove the lower aqueous layer. Note: the upper ether layer can be discarded or recycled if desired. Now, wash this lower aqueous layer with one 150-millilter portion of diethyl ether, and then place the mixture into a seperatory funnel, and recover the lower aqueous layer. Once again, the upper ether layer can be discarded or recycled if desired. Then place the collected lower aqueous layer into a suitable sized beaker, and then add to it, 150 grams of potassium sodium tartrate, and then rapidly stir the entire mixture for about 30 minutes. Now, slowly add to this mixture, 65 grams of sodium hydroxide, in small portions at a time, and then stir the entire alkaline mixture for about 1 hour. After stirring the alkaline mixture for about 1 hour, extract the entire alkaline mixture with three 150-milliliter portions of methylene chloride. After the extraction process, combine all methylene chloride portions (if not already done so), and then dry this combined methylene chloride portion by adding to it, 20 grams of anhydrous sodium sulfate. Then stir the entire mixture for about 10 minutes, and then filter-off the sodium sulfate. Thereafter, place the filtered methylene chloride portion into a distillation apparatus, and distil-off the methylene chloride at 40 Celsius. When no more methylene chloride passes over or is removed, stop the distillation process, and recover the left over remaining residue (after it has cooled). Finally, dissolve this left over residue into 30 milliliters of 99% isopropyl alcohol—after adding the residue to the alcohol, simply stir it for about 10 to 15 minutes. Thereafter, filter the alcohol mixture to remove any insoluble materials, and then place this alcohol mixture into an ice bath, and chill to 0 Celsius. When the temperature of the alcohol mixture reaches 0 Celsius, bubble into the mixture, 10 grams of dry hydrogen chloride gas (excess). After the addition of the hydrogen chloride, allow the alcohol mixture to stand at 0 Celsius for about 1 hour. Finally, filter-off the precipitated crystals, and then vacuum dry or air-dry them. Note: additional crystals of desired product can be obtained by distilling-off the alcohol, but only to the point where 80% of the total volume is reduced. Thereafter, filter the resulting alcohol concentrate to obtain the additional crystals. The result will be about 6 grams of the desired MESCALIN product

Note: Other salts of the freebase MESCALINE such as the sulfate dihydrate, tartrate, citrate, and phosphate can be prepared by adding the corresponding acid to the alcohol mixture of the freebase MESCALINE compound obtained at the end of step 2. For sulfuric acid or tartaric acid, 2 grams of 98% sulfuric acid or 3 grams of d-tartaric acid should be added to the freebase

MESCALINE in the alcohol mixture. For citric acid or phosphoric acid, 3 grams of citric acid or 2 grams of phosphoric acid should be added to the freebase MESCALINE in the alcohol mixture. The alcohol mixture before treatment with any of the aforementioned acids, should be chilled in an ice bath at 0 Celsius prior to addition. After treatment with the corresponding acid in each of these cases, the mixture can be filtered to recover the precipitated crystals. The crystals should then be vacuum dried or air-dried. Note: additional crystals of desired product can be obtained by distilling-off the alcohol, but only to the point where 80% of the total volume is reduced. Thereafter, filter the resulting alcohol concentrate to obtain the additional crystals. All the salts of MESCALINE are powerful stimulants/hallucinogens. For purified freebase MESCALINE, the regular hydrochloride salt should be treated with a 25% sodium carbonate (or sodium hydroxide) solution with stirring to liberate the freebase MESCALINE, which can then be extracted into 99% isopropyl alcohol, and the alcohol solvent then removed to leave behind the purified freebase MESCALINE. Note: the sulfate dihydrate salts melts at 183 to 186 Celsius, and was formerly the most common MESCALINE salt used.

Procedure B: Extraction of MESCALINE from San Pedro or peyote cactus

Procedure:

Personnel notes for procedure B: MESCALINE

Into a suitable beaker, or stainless steel container, place 500 grams of dry finely divided cactus (either san pedro or peyote), and then add in 500 milliliters of water. Note: if the cactus is fresh, it needs to be cut up into small pieces, and then chopped up in a puree machine or similar blender before use—in this case, use 1 kilogram of fresh cactus. If desired, you can dry your cactus instead of blending it fresh. Once the cactus has been placed into the suitable container, and thereafter water being added, bring the water to a boil, and just before the water boils, add in 30 milliliters of 35 to 38% hydrochloric acid (muriatic acid will work). Thereafter, boil the slightly acidic mixture for about 1 hour. After boiling the mixture for about 1 hour, remove the heat source and allow the mixture to cool to room temperature. Thereafter, place 500 milliliters of fresh water into a new clean beaker or stainless steel container, and then add in 30 milliliters of 35 to 38% hydrochloric acid (muriatic acid will work). Then filter-off the insoluble tuff or materials (from the previous extraction), and then place this filtered-off tuff into the new clean beaker filled with the fresh water. Thereafter, boil the entire mixture for about 1 hour (just like before). Note: after filtering-off the tuff, do not discard the water mixture. After boiling the tuff in water and dilute acid, once again, remove the heat source, and allow the mixture to cool to room temperature. Thereafter, filter-off the insoluble tuff, and then place this tuff into yet another new clean beaker or stainless steel container. Note: this second portion of water can be combined with the first portion. Thereafter, add to the tuff in the suitable container, 500 milliliters of fresh water, followed by 30 milliliters of 35 to 38% hydrochloric acid, and then boil the entire mixture for 1 hour. After 1 hour, remove the heat source, and allow the mixture to cool to room temperature. Thereafter, filter-off the tuff (and then finally discard it this time), and then combine the filtered water mixture with the previous 2. Finally, boil this combined water extract portion until the total volume equals 750 milliliters. When this point is achieved, stop the boiling process, and allow the mixture to cool to room temperature.

Now to the 750 milliliters of remaining aqueous liquid, add in 150 milliliters of 35 to 38% hydrochloric acid (muriatic acid will work), and then rapidly stir the entire mixture for about 1 hour at room temperature. Then extract this entire acidic mixture with three 75-milliliter portions of xylene (toluene should work with satisfactory results), and after each extraction process, keep the lower water layer, and discard any other layers. Note: the xylene or toluene layers will be the upper layers, and can be recycled if desired. After the extraction process, combine all lower aqueous portions, if not already done so. Then repeat this entire extraction process (using xylene or toluene) two more times, keeping the lower aqueous layer each time. The xylene or toluene upper layers can be recycled if desired after each extraction. After each successive extraction, combine all lower aqueous portions (if not already done so). After extraction of the water mixture three times, boil-off the water until only 500 milliliters in volume remains. When this is the result, remove the heat source, and allow the mixture to cool to room temperature.

To your 500 milliliters of remaining water mixture, add to it, 500 grams of a 10% sodium hydroxide solution prepared by adding and dissolving 50 grams of sodium hydroxide into 450 milliliters of water. Note: sodium hydroxide generates excessive heat when dissolved in water, so allow the alkaline mixture to cool before using. After the addition of the sodium hydroxide solution, continue to stir the water mixture for about 30 minutes at room temperature. Thereafter, extract this alkaline mixture with three 75-milliliter portions of xylene (toluene should work just as well), and after the extraction process, combine all xylene or toluene portions (if not already done so), and then dry this combined xylene portion by adding to it, 15 grams of anhydrous magnesium sulfate. Note: during the extraction process, the xylene will be the upper layer each time. After adding

the anhydrous magnesium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Finally, place this dried xylene or toluene portion into an ice bath, and then bubble into the mixture anywhere from 5 to 20 grams of dry hydrogen chloride. Note: during the addition of the hydrogen chloride, the MESCALINE and small amount of MESCALINE derivatives will precipitate. Continue bubbling the hydrogen chloride into the xylene or toluene mixture until no more precipitation takes place—this may take anywhere from 5 to 20 grams of hydrogen chloride. When no more precipitation takes place, stop bubbling in the hydrogen chloride, and then stir the xylene or toluene mixture for about 1 hour at 0 celsius. Thereafter, filter-off the precipitated salts, and then vacuum dry or air-dry them.

Now, take your died crystals, and place them into a suitable sized beaker. Thereafter, add in a sodium hydroxide solution prepared by adding and dissolving 30 grams of sodium hydroxide into 350 milliliters or water. Note: sodium hydroxide generates excessive heat when dissolved in water, so allow the alkaline mixture to cool before using. After the addition of the sodium hydroxide solution, rapidly stir the entire mixture for about 30 minutes at room temperature. Thereafter, briefly extract the alkaline mixture with two 20-milliliter portions of diethyl ether, and after the extraction process, discard or recycle the ether portions. Note: after each extraction process, the ether will be the upper layer each time. After the extraction process, extract the recovered lower aqueous mixture with three 75-milliliter portions of methylene chloride, and after the extraction process, combine all methylene chloride portions (if not already done so), and then dry this combine methylene chloride portion by adding to it, 10 grams of anhydrous magnesium sulfate. Note: after the extraction, the methylene chloride will be the bottom layer each time. After the addition of the magnesium sulfate, stir the entire mixture for about 10 minutes, and thereafter, filter-off the magnesium sulfate. Afterwards, place the filtered combined methylene chloride portion into a distillation apparatus, and distill-off the methylene chloride. When no more methylene chloride passes over or is collected, remove the left over remaining residue (after it has cooled), and then place it into a clean beaker. Now, add in about 90 milliliters of 99% isopropyl alcohol, and then stir the entire mixture to dissolve the bulk of the residue. Thereafter, quickly filter the mixture to remove any insoluble materials, and then place this isopropyl alcohol solution into an ice bath, and chill to 0 Celsius. Thereafter, bubble into this isopropyl alcohol solution, 5 to 10 grams of dry hydrogen chloride gas. After the addition of the hydrogen chloride, stir the isopropyl alcohol solution for about 1 hour, and then place this isopropyl alcohol mixture into a distillation apparatus, and distill-off the isopropyl alcohol at 82 Celsius until only 80% of the total solutions volume has been removed. When this is the case, stop the distillation process, and allow the left over remaining contents to cool to room temperature before removing from the distillation apparatus. Then filter the cooled left over alcohol concentrate to recover any insoluble solids. Thereafter, vacuum dry or air-dry these filtered-off solids. Note: the resulting filtered-off solids will be composed predominantly of MESCALINE ranging from 50 to 70% by weight. The remaining solids will consist of MESCALINE derivatives, and other alkaloids. The dosage in this case, should range from 300 to 400 milligrams per person to compensate for the lower concentration of the MESCALINE within the dry solids.

Note: Other salts of the freebase MESCALINE such as the sulfate dihydrate, tartrate, citrate, and phosphate can be prepared by adding the corresponding acid to the alcohol mixture of the freebase MESCALINE compound obtained at the end of step 2. For sulfuric acid or tartaric acid, 1 mole of sulfuric acid or d-tartaric acid should be added to 2 moles of the freebase MESCALINE in the alcohol mixture. For citric acid or phosphoric acid, 1 mole of citric acid or phosphoric acid should be added to 3 moles of the freebase MESCALINE in the alcohol mixture. The alcohol mixture before treatment with any of the aforementioned acids, should be chilled in an ice bath at 0 Celsius prior to addition (just like in step 2). After treatment with the corresponding acid in each of these cases, the alcohol mixture should be distilled to remove the alcohol, but only to the point where 80% of the total volume is reduced. Thereafter, filter the resulting alcohol concentrate to obtain the crystals.

Procedure C: Preparation of MESCALINE (cyanide process)

Materials:

1. 64 milliliters of a 33% sodium bisulfite solution	9. 10 grams of anhydrous magnesium sulfate
2. 5.8 grams of 3,4,5-trimethoxybenzaldehyde (see intermediate-0019. 3,4,5-TMB)	10. 2 milliliters of 98% sulfuric acid
3. 200 milliliters of 95% ethyl alcohol	11. 700 milligrams of palladium black catalyst
4. 4 grams of potassium cyanide	12. 115 milligrams of dry hydrogen gas
5. 25 milliliters of acetic anhydride	13. 30 grams of anhydrous sodium carbonate
6. 225 milliliters of diethyl ether	14. 30 milliliters of 99% isopropyl alcohol
7. 50 milliliters of a 10% sodium carbonate solution	15. 10 grams of dry hydrogen chloride gas
8. 50 milliliters of a 20% sodium bisulfite solution	

Summary: MESCALINE can be prepared in a modified process starting with the formation of 3,4,5-trimethoxybenzaldehyde cyanohydrin. This cyanohydrin is prepared by simply condensing 3,4,5-trimethoxybenzaldehye with sodium bisulfite to form a sodium bisulfite addition salt. This addition salt is then broken down into the cyanohydrin by reaction with a cyanide solution. The reaction is rather mild, and the desired product is easily recovered by filtration. The filtered-off solid is then washed, and

then dried in a desiccator until use. The 3,4,5-trimethoxybenzaldehyde cyanohydrin is then converted into 3,4,5-trimethoxybenzaldehyde cyanohydrin acetate by reaction with acetic anhydride under reflux conditions. The reflux period is rather moderate, and after a specified amount of time, the reaction mixture is cooled, and then evaporated. The left over residue is then taken-up into ether, and the ether mixture is then washed, dried, and then evaporated in the usual manner. The remaining residue, composed predominantly of the desired 3,4,5-trimethoxybenzaldehyde cyanohydrin acetate is then recrystallized from ethyl alcohol. The purified crystals of the 4,5-trimethoxybenzaldehyde cyanohydrin acetate are then converted into MESCALINE by reduction with hydrogen gas in the presence of a small amount of palladium black catalyst. After the calculated amount of hydrogen gas has been absorbed, the reaction is stopped, and the resulting reaction mixture is then basified with sodium carbonate. The basified reaction mixture is then evaporated to remove the alcohol, and the resulting residue is then taken-up into ether. The ether mixture is then filtered, and then evaporated under the usual manner. The left over residue is then dissolved into isopropyl alcohol and the desired MESCALINE is then precipitated by the addition of hydrogen chloride in the usual manner.

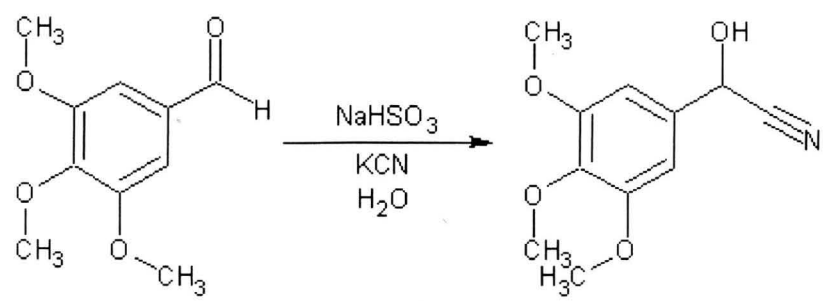
Hazards: Extinguish all flames before using diethyl ether, which is highly volatile and capable of forming explosive mixtures with air. Wear gloves when handling sulfuric acid, acetic anhydride and hydrogen chloride—use proper ventilation when handling hydrogen chloride, and avoid inhalation of the vapors. Potassium cyanide is a deadly poison, so wear gloves when handling and avoid skin absorption, ingestion, and inhalation of the dust. Hydrogen is highly explosive, so extinguish all flames before using, and avoid any sources of ignition.

Procedure:

Personnel notes for procedure C: MESCALINE

Step 1: Preparation of 3,4,5-trimethoxybenzaldehyde cyanohydrin

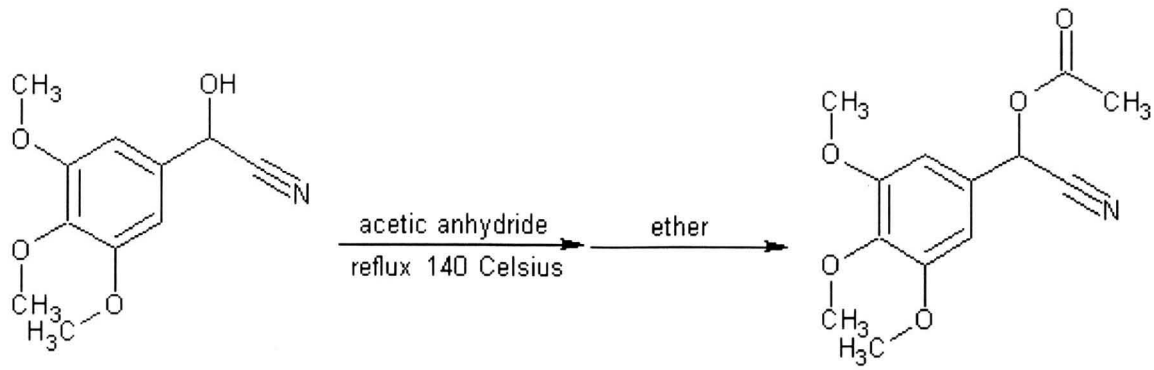
Into a suitable sized beaker or flask, equipped with motorized stirrer or other stiring means, place 14 milliliters of a 33% sodium bisulfite solution, and then gently warm this sodium bisulfite solution to about 30 to 35 Celsius. Thereafter, add in 5.8 grams of 3,4,5-trimethoxybenzaldehyde (see intermediate-0019. 3,4,5-TMB), and then stir the entire mixture until all solids dissolve. When the 3,4,5-trimethoxybenzaldehdye has dissolved, remove the heat source, and allow the mixture to cool to room temperature. Then stir the entire mixture for about 2 hours at room temperature, and then thereafter, allow the mixture to stand for 2 hours. Afterwards, filter-off the precipitated bisulfite addition product, and then wash this filtered-off addition product with two 25-milliliter portions of 95% ethyl alcohol, and then vacuum dry or air-dry the washed product. Thereafter, place this dried addition product into a clean beaker, and then add in 7 milliliters of water. Then stir the entire mixture to dissolve all solids. Then slowly add in, a cyanide solution, prepared by adding and dissolving 4 grams of potassium cyanide into 7 milliliters of water. During the addition of the cyanide solution, rapidly stir the aqueous solution of the addition product. After the addition of the cyanide solution, continue to stir the reaction mixture for about 1 hour at room temperature. Thereafter, place this reaction mixture into an ice bath, and chill to about 0 Celsius. Then allow this reaction mixture to stand at 0 Celsius for about 1 hour. After about 1 hour, filter-off the precipitated product, wash this product with one 50-millilter portion of a 33% sodium bisulfite solution, followed by one 50-millilter portion of cold water, and then place this washed precipitate into a desiccator filled with anhydrous sodium sulfate, and allow it to dry for about 24 hours, or until dry. Note: keep the resulting dried product in the desiccator until use. The result of the dried product will be about 6 grams with a melting point of 82 Celsius.



3,4,5-Trimethoxybenzaldehyde

Step 2: Preparation of 3,4,5-trimethoxybenzaldehyde cyanohydrin acetate

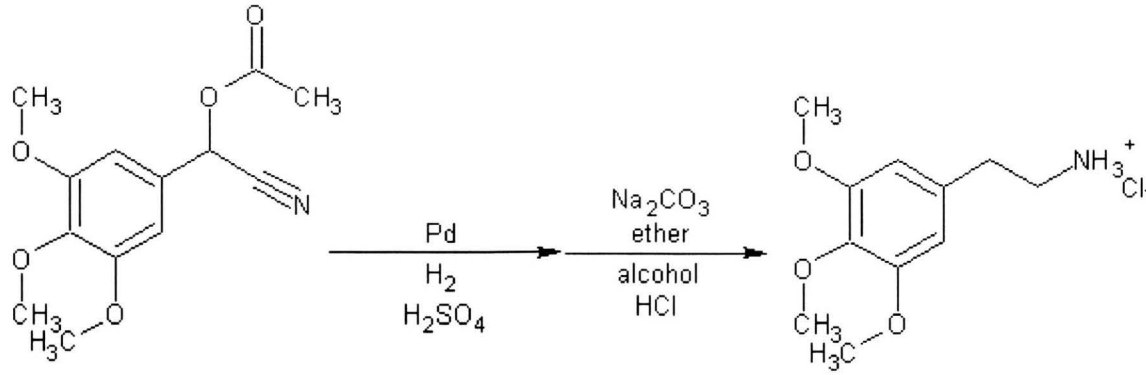
Into a standard reflux apparatus, place 5 grams of the product obtained in step 1, followed by 25 milliliters of acetic anhydride. Thereafter, reflux this entire mixture at about 140 Celsius for about 1 hour. After the reflux period, remove the heat source, and allow the refluxing reaction mixture to cool to room temperature. Thereafter, pour the entire reaction mixture into a shallow pan, and allow all liquids to evaporate-off. Note blowing air over the surface of the shallow pan using a conventional cooling fan can help speed up the evaporation process. Second note: vacuum distillation can be used to afford the recovery of the unreacted acetic anhydride, rather then just evaporating it off. Once all the liquid has been evaporated, recover the left over remaining residue, and then dissolve this recovered residue into 75 milliliters of diethyl ether. After adding the recovered residue to the diethyl ether, stir the entire mixture for about 30 minutes, and then quickly filter the reaction mixture to remove any potential insoluble materials (if any). Thereafter, wash this filtered ether mixture with one 50-milliliter portion of a 10% sodium carbonate solution (10% solution by weight based on the anhydrous sodium carbonate, not any of the hydrates), followed by one 50-millilter portion of a 20% sodium bisulfite solution, and then finally with one 50-milliliter portion of ice cold water. Note: after each washing, the ether will be the upper layer each time. After the washings, dry the washed ether portion by adding to it, 10 grams of anhydrous magnesium sulfate. Thereafter, stir this entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Finally, place the filtered ether mixture into a distillation apparatus, and distill-off the ether at 40 Celsius. When no more ether passes over or is collected, stop the distillation process, and collect the left over remaining residue (after it has cooled). Then dissolve this recovered residue into 75 milliliters of 95% ethyl alcohol, and then stir the entire mixture for about 10 minutes. Thereafter, filter-off any insoluble impurities (if any), and then recrystallize the desired dissolved product from this filtered 95% ethyl alcohol mixture, and after the recrystallization process, vacuum dry or air-dry the filtered-off crystals—then set the dried filtered-off crystals aside for step 3.



Step 3: Preparation of MESCALINE

Into a suitable flask, equipped with motorized stirrer or other stirring means, and gas inlet tube, place 5 grams of the product obtained in step 2, followed by 75 milliliters of 95% ethyl alcohol. Thereafter, stir the entire mixture to form a uniform mixture, and then add in 2 milliliters of 98% sulfuric acid, followed by 700 milligrams of palladium black catalyst. Thereafter, begin a slow stir, and then bubble into this reaction mixture, 115 milligrams of dry hydrogen gas. Note: see hydrogen in the reagents section for is preparation and assembly of a hydrogen generator. During the addition of the hydrogen gas, slowly stir the reaction mixture. After the addition of the hydrogen gas, slowly stir the entire reaction mixture at room temperature for about 1 hour, and then carefully filter-off the palladium black. Note: because palladium black is very expensive, the filtering process should be exercised with careful precision to recover every microgram of the palladium black, which can be dried, and then recycled over and over again. After removing the palladium black, place the reaction mixture into a suitable sized beaker, and then slowly and carefully add in 30 grams of anhydrous sodium carbonate, in small portions at a time. Note: during the addition of the sodium carbonate, moderately blend the filtered reaction mixture. After the addition of the sodium carbonate, stir the entire alkaline reaction mixture for about 1 hour, and then place the entire reaction mixture (without filtering) into a distillation apparatus, and distill-off the 95% ethyl alcohol at 78 Celsius. When no more ethyl alcohol passes over or is collected, stop the distillation process, and then recover the left over remaining oily residue (after it has cooled). Then place this collected left over residue into a clean beaker, and then add in 150 milliliters of diethyl ether, and then rapidly stir the entire mixture for about 1 hour. After 1 hour, filter the ether mixture to remove any insoluble impurities, and then place this ether mixture into a distillation apparatus, and distill-off the ether at 40 Celsius. When no more ether passes over or is collected, stop the distillation process, and then recover the left over remaining residue (after it has cooled). Finally, dissolve this left over residue (resinous material) into 30 milliliters of 99% isopropyl alcohol, and then stir the entire mixture for about 30 minutes. Thereafter, quickly filter the alcohol mixture to remove any insoluble materials (if any), and then place this filtered alcohol mixture into an ice bath, and chill to about 0 Celsius. Then bubble into this alcohol mixture, 10 grams of dry hydrogen chloride gas (excess), and after the addition, allow the entire alcoholic mixture to stand at 0 Celsius for about 1 hour. Then

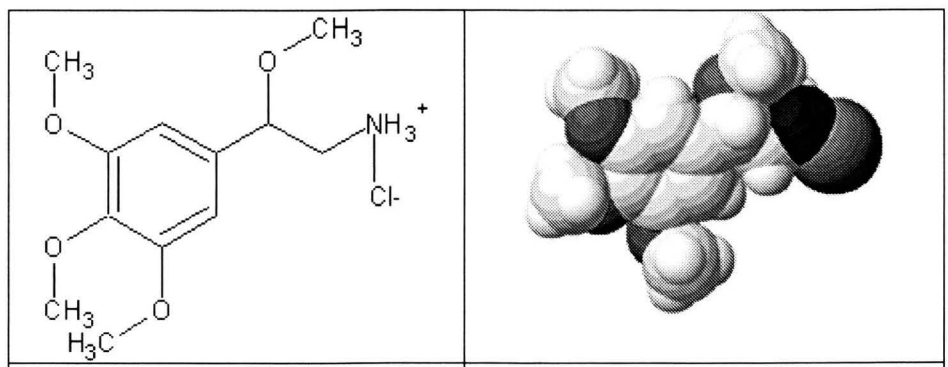
filter-off the precipitated crystals of the MESCALINE product, and then place the filtered alcohol mixture into a distillation apparatus, and distil-off the alcohol at 82 Celsius until about 80% of its total volume has been reduced. Thereafter, the alcohol concentrate should then be filtered to recover additional MESCALINE product. All recovered MESCALINE product should then be vacuum dried or air-dried.



MESCALINE

Note: Other salts of the freebase MESCALINE such as the sulfate dihydrate, tartrate, citrate, and phosphate can be prepared by adding the corresponding acid to the alcohol mixture of the freebase MESCALINE compound obtained at the end of step 3 in the usual manner.

0021. BOM. Beta-Methoxymescaline hydrochloride. 3,4,5-beta-tetramethoxyphenethylamine hydrochloride. *2-methoxy-2-(3,4,5-trimethoxyphenyl)ethanamine*



Structure	3D model

BOM forms colorless to white crystals, which may be cream colored or slightly pale when impure. The white crystals have a melting point of 199 Celsius. The crystals are soluble in water and alcohol, but insoluble in ether. BOM is a mild psychedelic amphetamine with general CNS stimulation. The exact nature of BOM has not been fully tested, but the small amount of research that has been conducted on this compound indicates it's a mild version of MESCALINE. It produces the usual stimulation effects, with a smaller degree of stimulation then amphetamine. The hallucinogenic effect of the drug is rather mild, and not fully tested on humans. The drug produces the usual psychedelic responses including enhancements of vision, colors, sights, sounds, ect., ect, but with a lesser degree then that of MESCALINE. Overall, BOM could be considered as a second rate psychedelic amphetamine; however, its total effects upon the human body have not fully been researched, and it might be possible that this substance has hidden potential in it. BOM has been studied when admixed with other drugs such as methamphetamine or other amphetamine derivatives with positive results—meaning that BOM could be admixed with amphetamines for enhanced feelings.

This substance is a controlled substance (psychedelic amphetamine) as listed in the US code of Federal regulations.

Toxicity: Low	**Rate of onset (average):** Moderate (may take up to 1 hour for effects to be realized)
Stimulation dosage (ingestion): 300 to 400 milligrams	**Duration of effects (average):** unknown
Stimulation dosage (inhalation): unknown	**Habit forming potential:** Low
Stimulation dosage (injection): unknown	**Estimated value U.S. (based on procedure):** $26 per gram.

	Street value cannot be calculated because its use is next to none (currently as of 2003).

Procedure A: Preparation of BOM

Materials:

1. 18 grams of beta-nitro-3,4,5-trimethoxystyrene (see procedure A of 0020. MESCALINE, step 1 for its preparation)	9. 80 milliliters of 99% isopropyl alcohol
2. 150 milliliters of anhydrous methyl alcohol	10. 150 milliliters of a 20% sodium hydroxide solution
3. 4 grams of metallic sodium	11. 250 grams of a 15% sulfuric acid solution
4. 60 milliliters of glacial acetic acid	12. 900 milliliters of methylene chloride
5. 300 milliliters of methyl alcohol	13. 50 grams of sodium hydroxide
6. 15 grams of lithium aluminum hydride	14. 20 grams of anhydrous magnesium sulfate
7. 300 milliliters of dry tetrahydrofuran (THF)	15. 10 grams of hydrogen chloride gas
8. 2.5 milliliters of 98% sulfuric acid	16. 150 milliliters of dry diethyl ether

Summary: BOM is readily prepared in a two step process starting with the formation of 1-methoxy-2-nitro-1-(3,4,5-trimethoxyphenyl)ethane. This intermediate is prepared by reacting beta-nitro-3,4,5-trimethoxystyrene with sodium methoxide. The sodium methoxide is generated on site by the addition of metallic sodium to methyl alcohol. The resulting 1-methoxy-2-nitro-1-(3,4,5-trimethoxyphenyl)ethane is then collected by precipitation by the addition of acetic acid and water. The precipitated crystals are then filtered-off, washed, and then dried. The dried crystals of 1-methoxy-2-nitro-1-(3,4,5-trimethoxyphenyl)ethane are then converted into BOM by reaction with lithium aluminum hydride in methanol. The reaction mixture is then refluxed, and the desired product then recovered in the usual manner.

Hazards: Use caution when handling metallic sodium, which is very dangerous, and should be kept away from water and most chemicals—wear gloves when handling. Wear gloves when handling lithium aluminum hydride, and avoid contact with water. Extinguish all flames before using diethyl ether, tetrahydrofuran, methyl alcohol, and isopropyl alcohol, all of which are flammable. Diethyl ether is capable of forming explosive mixtures with air. Methyl alcohol burns with a colorless flame, so burning methanol is hard to see. Avoid inhalation or ingestion of methyl alcohol, which is toxic. Wear gloves when handling glacial acetic acid, concentrated sulfuric acid, and sodium hydroxide, all of which are capable of producing skin irritation.

Procedure:

Personnel notes for procedure A: BOM

Step 1: Preparation of 1-methoxy-2-nitro-1-(3,4,5-trimethoxyphenyl)ethane

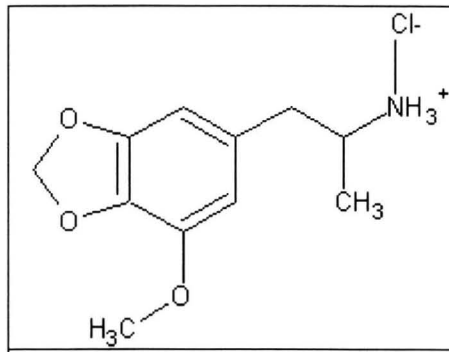
Into a suitable sized flask, equipped with thermometer, motorized stirrer or magnetic stirrer bar, and addition funnel, place 18 grams of beta-nitro-3,4,5-trimethoxystyrene (see procedure A of 0020. MESCALINE, step 1 for its preparation), followed by 100 milliliters of anhydrous methyl alcohol. Then prepare a solution by first, adding 50 milliliters of anhydrous methyl alcohol into a suitable beaker, and then chill this alcohol to 0 Celsius. Thereafter, slowly add in, 4 grams of metallic sodium, in small portions, over a period of time sufficient to keep the methyl alcohol mixture below 40 Celsius. After the addition of the metallic sodium, continue to stir the methyl alcohol (which will contain sodium methoxide) for about 30 minutes, or until the methyl alcohol mixture cools to room temperature. Thereafter, place this methyl alcohol solution (containing the sodium methoxide) into the addition funnel of the suitable flask, and thereafter, add this methyl alcohol solution (containing the sodium methoxide) to the methyl alcohol/beta-nitro-3,4,5-trimethoxystyrene mixture over a period of about 10 minutes while rapidly stirring the reaction mixture. After the addition, continue to stir the reaction mixture for about 10 minutes, and then add in 60 milliliters of glacial acetic acid. After the addition of the glacial acetic acid, continue to stir the reaction mixture for about 10 minutes, and immediately thereafter, add in 300 milliliters of water, and then continue stirring for about 30 minutes. After stirring for about 30 minutes, filter-off the precipitated white solids, and then wash them with three 50-milliliter portions of cold water, and then vacuum dry or air-dry these washed solids. Thereafter, recrystallize these dried solids from 300 milliliters of boiling methyl alcohol. After the recrystallization process, vacuum dry or air-dry the filtered-off solids. The result will be about 12 grams of the desired product as cream colored crystals with a melting point of 144 Celsius.

Step 2: Preparation of BOM

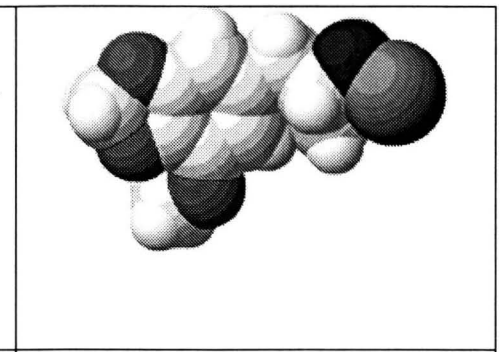
Into a suitable reflux apparatus, fitted with motorized stirrer, and addition funnel, place a solution prepared by adding and dissolving 15 grams of lithium aluminum hydride into 200 milliliters of dry tetrahydrofuran (THF). Note: place a calcium chloride drying tube on the top of the reflux condenser to keep moisture out. Thereafter, cool this lithium aluminum hydride mixture to about 0 Celsius using an ice bath, and when its temperature reaches about 0 Celsius, slowly add in 2.5 milliliters of 98% sulfuric acid—place this sulfuric acid into the addition funnel before adding. During the addition of the sulfuric acid, rapidly stir the lithium aluminum hydride/THF mixture. After the addition of the sulfuric acid, add 12 grams of the product obtained in step 1 (through the top of the reflux condenser), over a period of about 5 minutes. Note: simply remove the calcium chloride drying tube, and then place it back on immediately after the addition. Note: during the addition of the sulfuric acid and the product obtained in step 1, maintain the temperature of the reaction mixture below 5 Celsius with rapid stirring. After the addition, continue to stir the reaction mixture for about 10 minutes at a temperature below 5 Celsius. Then, reflux the entire reaction mixture at 68 Celsius for about 4 hours. After 4 hours, remove the heat source, and allow the reaction mixture to cool to room temperature. Thereafter, add to the reaction mixture, 20 milliliters of 99% isopropyl alcohol, followed by 150 milliliters of a 20% sodium hydroxide solution. Note: during both additions, rapidly stir the reaction mixture, and maintain its temperature below 40 Celsius at all times. After the additions, filter the alkaline reaction mixture to remove any insoluble impurities, and then briefly wash the filtered-off solids with two 50-milliliter portions of tetrahydrofuran, and then combine these two tetrahydrofuran washing portions with the filtered reaction mixture. Then, place this combined filtered reaction mixture into a distillation apparatus, and distill-off the tetrahydrofuran at 68 Celsius until no more tetrahydrofuran passes over, or is collected. Once this stage has been reached, recover the left over remaining reddish-brown oil (after it has cooled), and then place it into a suitable sized beaker. Then carefully add to this reddish-brown oil, 250 grams of a 15% sulfuric acid solution. During the addition of the sulfuric acid, rapidly stir the mixture, and maintain its temperature below 40 Celsius. After the addition of the sulfuric acid, rapidly stir the entire mixture for about 30 minutes. Thereafter, briefly extract this entire acidic mixture with three 150-milliliter portions of methylene chloride, and after the extraction process, discard or recycle the methylene chloride portions. Note: the methylene chloride will be the upper layer each time after each extraction. Now, to the recovered lower acidic aqueous layer, very slowly add in, 300 grams of a 16% sodium hydroxide solution prepared by adding and dissolving 50 grams of sodium hydroxide into 250 milliliters of water. Note: sodium hydroxide generates excessive heat when dissolved in water, so allow the alkaline solution to cool before using. During the addition of the sodium hydroxide, rapidly stir the mixture. After the addition of the sodium hydroxide, continue to stir the mixture for about 30 minutes, and thereafter, extract the entire alkaline mixture with three 150-milliliter portions of methylene chloride. After the extraction process, combine all methylene chloride portions, if not already done so, and then dry this combined methylene chloride portion, by adding to it, 20 grams of anhydrous magnesium sulfate. Note: after each extraction process, the methylene chloride will be the upper layer each time. After the addition of the magnesium sulfate, stir the entire mixture for about 10 minutes, and thereafter, filter-off the magnesium sulfate. Thereafter, place the filtered dried methylene chloride portion into a distillation apparatus, and distill-off the methylene chloride at 40 Celsius until no more methylene chloride passes over or is collected. When this is the case, stop the distillation process, and recover the left over remaining residue (after it has cooled), and then place it into a suitable sized beaker. Thereafter, add in 60 milliliters of 99% isopropyl alcohol, and then stir the entire mixture to dissolve the bulk of the residue. Then quickly filter this alcohol mixture to remove any insoluble materials. Finally, place this filtered alcohol mixture into an ice bath, and chill to about 0 Celsius. Finally, bubble into the alcohol mixture, 10 grams (excess) of dry hydrogen chloride gas. After the addition of the hydrogen chloride, stir the entire alcohol mixture for about 30 minutes at 0 Celsius. Then add in 150 milliliters of dry diethyl ether, and then stir the resulting mixture for about 30 minutes. Then filter-off the precipitated product, and then vacuum dry or air-dry it. The result will be about 5 grams of the desired product.

Note: As mentioned many times in this book, other salts of the freebase compound can be obtained by treating the freebase solution with the desired acid. Other salts of the freebase BOM such as the sulfate, tartrate, citrate, and phosphate can be prepared by adding the corresponding acid to the alcohol mixture of the freebase BOM compound obtained at the end of step 2. For sulfuric acid or tartaric acid, the usual mole quantity should be added to the usual mole quantity of the freebase BOM which is dissolved in the alcohol. Note: this means 1 mole of acid per 2 moles of the BOM on a dry bases—not the total weight of the alcohol mixture. For citric acid or phosphoric acid, the usual mole quantity (1 mole) of citric acid or phosphoric acid should be added to the usual mole quantity of the freebase BOM (3 moles) in the alcohol mixture. The alcohol mixture before treatment with any of the aforementioned acids, should be chilled in an ice bath at 0 Celsius prior to addition. After treatment with the corresponding acid in each of these cases, the alcohol mixture can be treated with 2 volumes (or slight larger then) of diethyl ether. Thereafter, the solvent mixture can be filtered to recover the precipitated crystals. The crystals should then be vacuum dried or air-dried. As expected, all salts of BOM have similar properties.

0022. MMDA. 3-Methoxy-4,5-methylenedioxyamphetamine hydrochloride. *1-(7-methoxy-1,3-benzodioxol-5-yl)propan-2-amine hydrochloride*



Structure



3D model

MMDA is a characteristic psychedelic amphetamine with similar effects to MDMA, but with more potency towards hallucinations. MMDA is currently ranked low in essence of street consumption, primarily due to lack of information on its preparation, but it definitely shows a future appearance as a major street drug. This is primarily due to its hallucinogenic activity, which is only slightly distracted by its amphetamine like stimulation effects. MMDA appears to produce significant hallucinogenic activity when users have their eyes closed. Many users have described such action as uncontrollable or rather simplistic moving pictures described by many as "movies". During normal activity, such as everyday activities, the drug produces the usual hallucinogenic effects similar to LSD and MESCALINE with the usual enhancements to sight, smell, sound, feelings, and touch. Other effects include the usual stimulation resembling amphetamine, but with lesser "upper" feelings. In essence, MMDA is kind of a combination of LSD, and MESCALINE combined with the "upper" sensations of the amphetamine class. Overall, users of this compound have stated it to be a wonderful trip with few side effects, and absolutely no withdrawal symptoms—users after coming off the drug experience intense feelings of satisfaction and happiness.

This substance is a controlled substance (psychedelic amphetamine) as listed in the US code of Federal regulations.

Toxicity: Low	**Rate of onset (average):** Moderate (may take up to 1 hour for effects to be realized)
Stimulation dosage (ingestion): 100 to 250 milligrams	**Duration of effects (average):** unknown (tends to fluctuate)
Stimulation dosage (inhalation): 80 to 150	**Habit forming potential:** Low
Stimulation dosage (injection): 50 to 100 (estimated)	**Estimated value U.S. (based on procedure):** $30 per gram

Procedure A: Preparation of MMDA

Materials:

1. 150 milliliters of dimethylformamide (DMF)	14. 200 milliliters of a 5% sulfuric acid solution
2. 8.4 grams of 5-hydroxyvanillin (obtained in step 3 of procedure B of Intermediate-0019. 3,4,5-TMB)	15. 17 grams of anhydrous sodium carbonate
3. 29 grams of potassium fluoride	16. 15 grams of anhydrous sodium sulfate
4. 4.6 grams of methylene chloride	17. 10 grams of dry hydrogen chloride
5. 640 milliliters of diethyl ether	18. or 20 grams of picric acid
6. 225 milliliters of a 10% sodium carbonate solution	19. or 295 milliliters of 95% ethyl alcohol
7. 15 grams of anhydrous sodium sulfate	20. or 30 milliliters of a 10% sodium hydroxide solution
8. 18 milliliters of glacial acetic acid	21. or 50 milliliters of a 5% sodium hydroxide solution
9. 2.7 milliliters of nitroethane	22. or 75 milliliters of methylene chloride
10. 1.6 grams of anhydrous ammonium acetate	23. or 10 grams of anhydrous magnesium sulfate
11. 50 milliliters of a 10% acetic acid solution	24. or 150 milliliters of dry diethyl ether
12. 30 milliliters of boiling methyl alcohol	25. or 5 grams of hydrogen chloride gas
13. 3.7 grams of lithium aluminum hydride	

Summary: MMDA can be prepared in a three-step process starting with myristicinaldehyde. This intermediate is prepared by reacting 5-hydroxyvanillin with potassium fluoride in the presence of methylene chloride under reflux. After the reaction, the reaction mixture is cooled, extracted with ether, and the resulting ether extracts are then treated in the usual manner. Upon evaporation of the ether, followed by recrystallization, the desired myristicinaldehyde is obtained. This compound is then converted into 2-nitro-isomyristicin by condensation with nitroethane. The reaction is very similar to most nitro condensations in this book, and after the reaction, the desired product is obtained in the usual manner. The corresponding nitro product is then converted into the desired MMDA by reduction with lithium aluminum hydride in the presence of diethyl ether. After the reduction, the reaction mixture is acidified (to remove lithium and aluminum impurities), and then basified by the addition of

sodium carbonate. Thereafter, the reaction mixture is extracted with ether, and the ether is then treated with hydrogen chloride in the usual manner to obtain the desired product. Instead of extracting the basified reaction mixture with ether, the basified reaction mixture can be treated with picric acid to form the insoluble picric salt of MMDA. The picric salt is easily recovered by filtration, whereby it is broken down into the freebase MMDA and picric acid by the addition of sodium hydroxide. The freebase is extracted into methylene chloride, and the methylene chloride is then removed under the usual manner. The left over residue is then dissolved into ether, and the desired MMDA product is then precipitated in the usual manner by the addition of hydrogen chloride gas.

Hazards: Extinguish all flames before using diethyl ether, which is highly flammable and capable of forming explosive mixtures with air. Extinguish all sources of ignition before using ethyl alcohol, methyl alcohol, and nitroethane. Nitroethane is highly flammable, so use caution. Methyl alcohol burns with a colorless flame, so burning alcohol cannot be seen. Wear gloves when handling sodium hydroxide, concentrated hydrochloric acid, and glacial acetic acid. Lithium aluminum hydride reacts violently with water, so keep in airtight bottles in a cool place.

Procedure:

Personnel notes for procedure A: MMDA

Step 1: Preparation of Myristicinaldehyde

Into a suitable flask or beaker, equipped with motorized stirrer or magnetic stirrer, place 150 milliliters of dimethylformamide (DMF), followed by 8.4 grams of 5-hydroxyvanillin (obtained in step 3 of procedure B of Intermediate-0019. 3,4,5-TMB). Then briefly stir the entire mixture to dissolve all solids. Thereafter, gradually add in 29 grams of potassium fluoride, and then rapidly stir the entire reaction mixture for about 1 hour. Note: if after 1 hour, the reaction mixture is still warm, continue to stir the reaction mixture until it cools to room temperature. Thereafter, add in 4.6 grams of methylene chloride, and then place the entire reaction mixture into a reflux apparatus, and reflux at 120 Celsius for 1 hour. After refluxing the reaction mixture at 120 Celsius for 1 hour, remove the heat source, and allow the reaction mixture to cool to room temperature. Now, extract the entire reaction mixture with three 50-milliliter portions of diethyl ether, and after the extraction period, combine all ether extracts (if not already done so), and then wash this combined ether portion with three 25-milliliter portions of cold water. Note: after the extraction and washing portions, the ether will be the upper layer each time. After the extraction and water washing portions, wash the ether portion with three 75-milliliter portions of a 10% sodium carbonate solution (based by weight on the anhydrous sodium carbonate in water, not any of the hydrates). After each washing portion, the ether will be the upper layer each time. After the washing of the ether portion with sodium carbonate solution, dry the ether portion by adding to it, 15 grams of anhydrous sodium sulfate, and then stir the entire mixture for about 10 minutes. Finally, filter-off the sodium sulfate, and then place the filtered ether portion into a distillation apparatus, and distill-off the diethyl ether. After the diethyl ether has been completely removed by distillation, recover the left over remaining residue (after it has cooled), and then recrystallize it from 100 milliliters of dry hexane. After the recrystallization process, vacuum dry or air-dry the filtered-off crystals.

Step 2: Preparation of 2-nitro-isomyristicin

Into a suitable beaker equipped with motorized stirrer or magnetic stirrer, place 5 grams of the product obtained in step 1, followed by 18 milliliters of glacial acetic acid. Then stir the entire mixture for about 10 minutes, and then add in, 2.7 milliliters of nitroethane, followed by 1.6 grams of anhydrous ammonium acetate. Thereafter, place the entire mixture into a reflux apparatus, and reflux the entire mixture for about 1 hour at 100 Celsius. Thereafter. Remove the heat source, and allow the reaction mixture to cool to room temperature. Then add in, 50 milliliters of ice-cold water, and then stir the entire mixture for about 30 minutes. Thereafter, place the entire mixture into an ice bath, and chill to about 0 Celsius. Then allow the mixture to stand at 0 Celsius for several hours. Thereafter, filter-off the precipitated product, wash with two 25-milliliter portions of an ice-cold 10% acetic acid solution (pre-chilled in a freezer), and then vacuum dry or air-dry the solids. Finally, recrystallize the dry filtered-off solids from 30 milliliters of boiling methyl alcohol, and after the recrystallization process, vacuum dry or air-dry the filtered-off solids.

Step 3: Preparation of MMDA (method 1)

Into a suitable 3-neck flask, equipped with motorized stirrer, thermometer, reflux condenser (fitted with a calcium chloride drying tube attached thereto), and addition funnel, place 3.7 grams of lithium aluminum hydride, immediately followed by 100

milliliters of dry diethyl ether. Then, briefly stir the mixture for a few minutes, and then prepare a solution by adding and dissolving 5 grams of the product obtained in step 2, into 200 milliliters of diethyl ether. Thereafter, place this solution into the addition funnel, and then begin to reflux the lithium aluminum hydride solution at 40 to 50 Celsius, and when the lithium aluminum hydride mixture reaches 40 to 50 Celsius, gradually add, drop-wise, the ether solution of the product obtained in step 2 (contained in the addition funnel), over a period of time sufficient to keep the reaction mixture around 40 to 50 Celsius. During the addition, rapidly stir the reaction mixture. After the addition, continue to reflux the reaction mixture at 40 to 50 Celsius for 2.5 hours with rapid stirring. Thereafter, remove the heat source, and allow the reaction mixture to cool to room temperature. Then slowly pour the entire reaction mixture into 200 milliliters of a 5% sulfuric acid solution, and then stir the entire mixture for about 10 minutes. Then place the entire mixture into a seperatory funnel, and then remove the lower aqueous acidic layer. Note: the upper ether layer can be discarded or recycled if desired. Now, slowly add to the recovered lower aqueous layer, a sodium carbonate solution prepared by adding and dissolving 17 grams of anhydrous sodium carbonate into 80 milliliters of cold water (note: if crystals of the hydrate form when the sodium carbonate is added to the water, dissolve these crystals into the bulk of the solution). After the addition of the sodium carbonate solution, immediately heat this alkaline mixture to about 80 Celsius with stirring for about 30 minutes. Thereafter, remove the heat source, and allow the alkaline mixture to cool to room temperature. Thereafter, filter-off any insoluble materials, and then extract this entire alkaline mixture with three 50-millilter portions of diethyl ether. After the extraction process, combine all ether portions (if not already done so), and then dry this combined ether potion by adding to it, 15 grams of anhydrous sodium sulfate, and then stir the entire mixture for about 10 minutes—thereafter, filter-off the sodium sulfate. Then place this filtered ether portion into an ice bath, and chill to about 0 Celsius. Thereafter, bubble into the mixture, 5 grams of dry hydrogen chloride gas (excess), and after the addition, allow the entire mixture to stand at 0 Celsius for about 2 hours. After 2 hours, filter-off the precipitated crystals of the desired MMDA, wash them with two 20-milliliter portions of diethyl ether, and thereafter, vacuum dry or air-dry the product. The result will be about 3 grams of the desired product with a melting point of 191 Celsius.

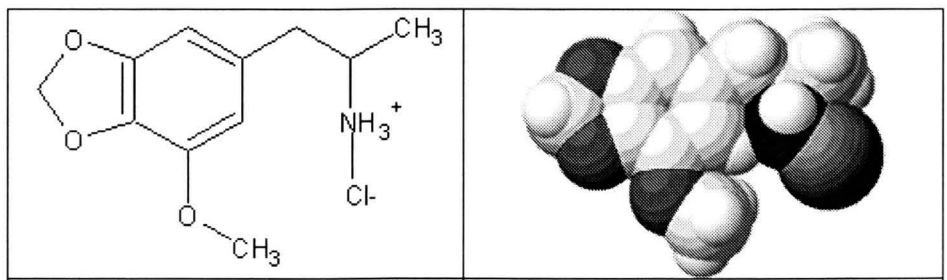
Step 3: Preparation of MMDA (method 2)

Into a suitable 3-neck flask, equipped with motorized stirrer, thermometer, reflux condenser (fitted with a calcium chloride drying tube attached thereto), and addition funnel, place 3.7 grams of lithium aluminum hydride, immediately followed by 100 milliliters of dry diethyl ether. Then, briefly stir the mixture for a few minutes, and then prepare a solution by adding and dissolving 5 grams of the product obtained in step 2 into 200 milliliters of diethyl ether. Thereafter, place this solution into the addition funnel, and then begin to reflux the lithium aluminum hydride solution at 40 to 50 Celsius, and when the lithium aluminum hydride mixture reaches 40 to 50 Celsius, gradually add, drop-wise, the ether solution of the product obtained in step 2 (contained in the addition funnel), over a period of time sufficient to keep the reaction mixture around 40 to 50 Celsius. During the addition, rapidly stir the reaction mixture. After the addition, continue to reflux the reaction mixture at 40 to 50 Celsius for 2.5 hours with rapid stirring. Thereafter, remove the heat source, and allow the reaction mixture to cool to room temperature. Then slowly pour the entire reaction mixture into 200 milliliters of a 5% sulfuric acid solution, and then stir the entire mixture for about 10 minutes. Then place the entire mixture into a seperatory funnel, and then remove the lower aqueous acidic layer. Note: the upper ether layer can be discarded or recycled if desired. Now, slowly add to the recovered lower aqueous layer, a sodium carbonate solution prepared by adding and dissolving 17 grams of anhydrous sodium carbonate into 80 milliliters of cold water (note: if crystals of the hydrate form after dissolving the sodium carbonate into the water, dissolve the crystals into the bulk of the solution). After the addition of the sodium carbonate solution, immediately heat this alkaline mixture to about 80 Celsius with stirring for about 30 minutes. After 30 minutes, quickly filter the mixture to remove any insoluble impurities, and then heat this hot filtered mixture to 100 Celsius, and then carefully add to it, a picric acid mixture, prepared by a) adding 20 grams of picric acid into 10 milliliters of cold water, and then mixing this mixture for about 10 minutes to form a slurry, and then b) adding this picric acid slurry to 220 milliliters of boiling 95% ethyl alcohol. After the addition of the picric acid mixture, rapidly stir the entire mixture for about 30 minutes, and then remove the heat source, and allow the mixture to cool to room temperature. Thereafter, place this cooled mixture into an ice bath, and let stand for about 1 hour. Thereafter, filter-off the precipitated picric acid salt of MMDA, wash with three 25-milliliter portions of 95% ethyl alcohol, and then vacuum dry or air-dry the crystals. Now, place the dried filtered-off crystals into a suitable sized beaker, and then add to it, 30 milliliters of a 10% sodium hydroxide solution. Then rapidly stir the entire mixture for about 30 minutes. After 30 minutes, quickly filter the alkaline mixture to remove insoluble impurities, and then add to the filtered mixture, 25 milliliters of water, followed by 50 milliliters of a 5% sodium hydroxide solution, and then rapidly stir the entire mixture for about 30 minutes. Then, extract this entire mixture with three 25-milliliter portions of methylene chloride, and after the extraction process, combine all methylene chloride portions (if not already done so), and then dry this combined methylene chloride portion by adding to it, 10 grams of anhydrous magnesium sulfate. Thereafter, stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Then place this filtered methylene chloride portion into a distillation apparatus, and remove the methylene chloride at 40 Celsius by distillation. When no more methylene chloride passes over or is collected, remove the left over remaining residue (after it has cooled), and then dissolve this left over residue into 100 milliliters of dry diethyl ether. Last but not least, place this ether mixture into an ice bath, and chill to 0 Celsius. Thereafter, bubble into this ether mixture, 5 grams of hydrogen chloride gas, and after the addition, allow the acidified ether mixture to

stand for 1 hour at 0 Celsius. Then filter-off the precipitated MMDA product, wash with two 25-millilter portions of diethyl ether, and then vacuum dry or air-dry the crystals. The result will be about 3 grams of the desired product.

Note: As mentioned many times in this book, other salts of the freebase compound can be obtained by treating the freebase solution with the desired acid. Other salts of the freebase MMDA such as the sulfate, tartrate, citrate, and phosphate can be prepared by adding the corresponding acid to the ether mixture of the freebase MMDA compound obtained at the end of step 3 (both of them). For sulfuric acid or tartaric acid, the usual mole quantity should be added to the usual mole quantity of the freebase MMDA which is dissolved in the ether. Note: as with all of these notes everywhere in this book, this means 1 mole of acid per 2 moles of the MMDA on a dry bases—not the total weight of the ether mixture. For citric acid or phosphoric acid, the usual mole quantity (1 mole) of citric acid or phosphoric acid should be added to the usual mole quantity of the freebase MMDA (3 moles) in the ether mixture. The ether mixture before treatment with any of the aforementioned acids, should be chilled in an ice bath at 0 Celsius prior to addition. After treatment with the corresponding acid in each of these cases, the ether mixture can be filtered to recover the precipitated crystals. The crystals should then be vacuum dried or air-dried. As expected, all salts of MMDA have similar properties. Note: the citrate salt may be more potent then the hydrochloride.

Procedure B: Preparation of racemic-MMDA



Structure	3D model

Materials:

1. 8.3 grams of iodine	8. 80 grams of ammonium sulfate or 35 grams of ammonium chloride
2. 500 milligrams of red phosphorus	9. 42 milliliters of methyl alcohol
3. 12 grams of myristicin (see intermediate-0026. Myristicin)	10. 48 grams of sodium hydroxide
4. 300 milliliters of methylene chloride	11. 100 milliliters of a 10% sodium hydroxide solution
5. 50 milliliters of a ice cold 5% sodium hydroxide solution	12. 15 grams of anhydrous magnesium sulfate
6. 100 milliliters of a ice cold 10% sulfuric acid solution	13. 150 milliliters of dry diethyl ether
7. 100 milliliters of a ice cold 5% baking soda solution	14. 15 grams of hydrogen chloride gas

Summary: racemic-MMDA can be prepared in a rather simple 2½ step process starting with the formation of myristicin iodide (iodinated myristicin). This iodinated myristicin can be prepared by treating myristicin with hydrogen iodide. The hydrogen iodide is generated on sight by the reaction of iodine with red phosphorus in the presence of water. After the iodination process, the iodinated myristicin is collected by washing the reaction mixture several times with various reagents, followed by evaporation of the solvent to recover the left over remaining iodinated myristicin as a residue. The residue is then treated with methyl alcohol/ammonia solution under mild heat and pressure for several days. The methyl alcohol/ammonia solution is prepared by first, generating ammonia gas by the addition of sodium hydroxide with an ammonium salt, and then bubbling the liberated ammonia gas into the methyl alcohol. After the reaction, the MMDA is collected in the usual manner—i.e., evaporation of the methyl alcohol, treatment of the left over residue with sodium hydroxide, followed by extraction with common solvent, removal of the solvent in the usual manner, followed by dissolving the residue into ether, and then finally precipitating the desired product by the addition of hydrogen chloride.

Hazards: Use maximum ventilation and extinguish all flames before using diethyl ether, which is highly flammable and capable of forming explosive mixtures with air—use extreme caution. Be sure to keep methyl alcohol away from fire and other sources of ignition, as methyl alcohol burns with a colorless flame. Wear gloves when handling iodine, and use good ventilation—iodine is very corrosive, and evolves irritating fumes. Wear gloves when handling sulfuric acid and sodium hydroxide.

Procedure:

Personnel notes for procedure B: racemic-MMDA

Into the apparatus illustrated in figure 009 (where indicated), place 8.3 grams of iodine, followed by 500 milligrams of red phosphorus, followed by 1.5 milliliters of water. Immediately thereafter, place 12 grams of myristicin where indicated, followed by 150 milliliters of methylene chloride, and then gently warm the reaction flask containing the iodine. As the flask is warmed, a gentle stream of hydrogen iodide will form, and pass over into the flask containing the myristicin. Note: during the addition of the hydrogen iodide into the myristicin/methylene chloride mixture, the myristicin mixture should be kept at 0 Celsius with rapid stirring. Note: if gently warming the iodine mixture does not work to produce a steady stream of hydrogen iodide, raise the temperature of the iodine mixture. Continue to feed the hydrogen iodide gas into the myristicin mixture until no more hydrogen iodide passes over. This point will be reached in about 30 to 45 minutes, but more time may be needed depending on purity of the iodine, and temperature. After the addition of the hydrogen iodide, quickly disassemble the apparatus illustrated in the figure, and then place the myristicin/methylene chloride mixture, which will now contain the iodinated myristicin, into a suitable beaker, and then wash this iodinated myristicin mixture with 50 milliliters of a ice cold 5% sodium hydroxide solution, followed by 50 milliliters of ice cold water, followed by 100 milliliters of a ice cold 10% sulfuric acid solution, followed by two 50-millilter portions of a ice cold 5% baking soda solution, followed by two 50-millilter portions of ice cold water. Note: after each washing portion, use a seperatory funnel to collect the solvent layer, which will be the upper layer each time. After the washing portions, place the collected upper solvent layer (containing the iodinated myristicin and methylene chloride) into a distillation apparatus, and remove the methylene chloride at 40 Celsius. When no more methylene chloride passes over or is collected, stop the distillation process, and recover the left over remaining residue (after it has cooled), and then set this iodinated myristicin aside for just a moment.

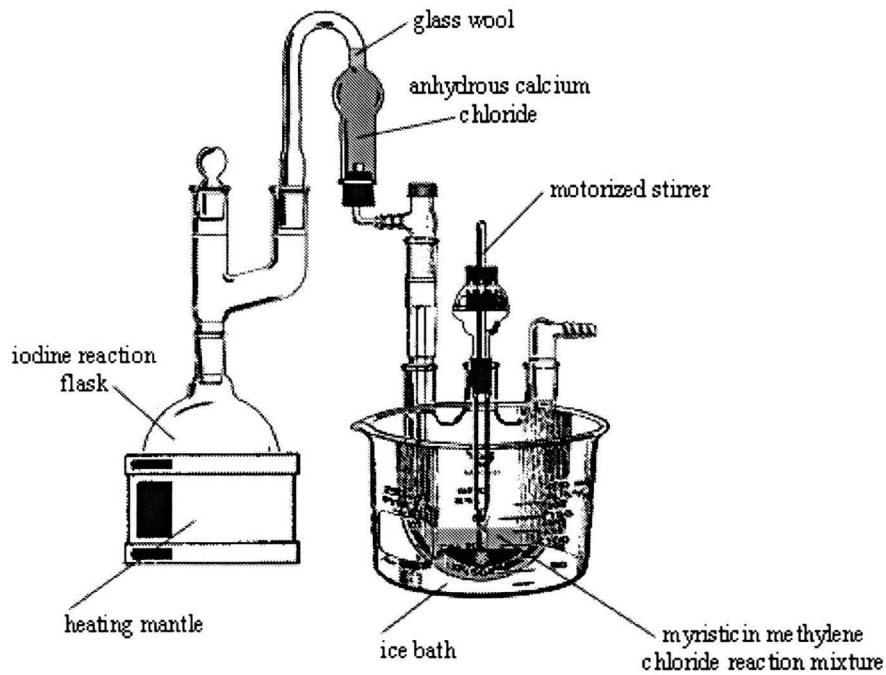


Figure 046. Set-up for the iodination of myristicin. Note: this set-up is merely a suggestive illustration, and other apparatus can be used, as long as they are similar in nature to the one illustrated above.

Now, set-up the apparatus in figure 000, and then place 80 grams of ammonium sulfate or 35 grams of ammonium chloride into the reaction flask. Then place 42 milliliters of methyl alcohol into the receiver flask where illustrated, and keep this methyl alcohol in an ice bath and chilled to 0 Celsius. Immediately thereafter, begin adding, drop-wise, a sodium hydroxide solution prepared by adding and dissolving 48 grams of sodium hydroxide into 50 milliliters of water. Note: sodium hydroxide generates excessive heat when dissolved in water, so allow the solution to cool before using. During the addition of the concentrated sodium hydroxide solution, ammonia gas will be steadily evolved, and will pass over into the receiver flask containing the methyl alcohol, forming an ammonia/methyl alcohol solution. Note: during the addition of the ammonia gas to the methyl alcohol, the methyl alcohol should be kept at 0 Celsius at all times. The addition should take no longer then 20 to 30

minutes—depending on how long you take to drip in the sodium hydroxide solution onto the ammonium salt in the reaction flask. When no more ammonia gas passes over, disassemble the apparatus, and place the ammonia/methyl alcohol solution into a suitable clean flask or beaker and set aside for just a moment.

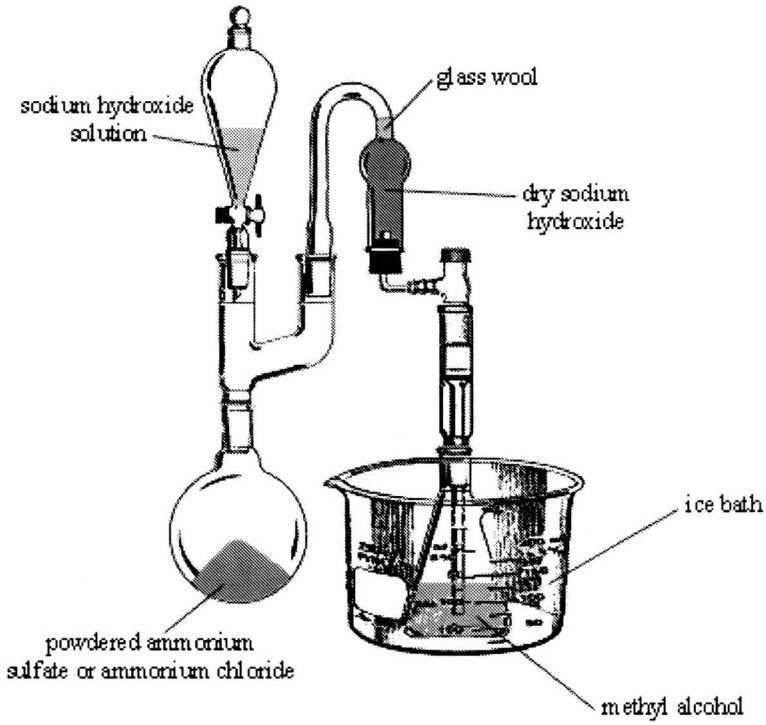
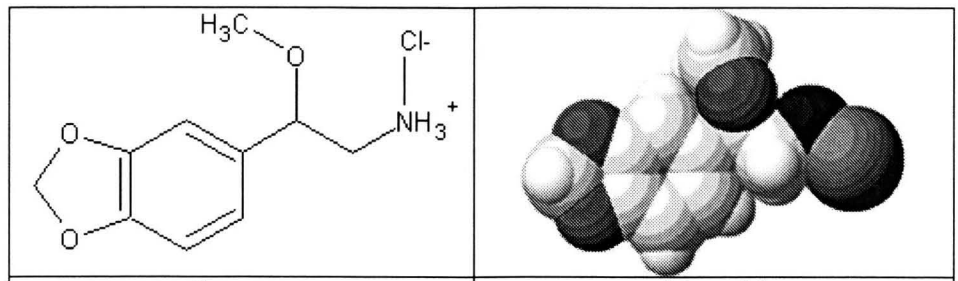


Figure 047. Set-up for preparing methyl alcohol/ammonia solution. Note: this set-up is merely a suggestive illustration, and other apparatus can be used, as long as they are similar in nature to the one illustrated above.

Now, place the iodinated myristicin residue (prepared in the first paragraph) into a suitable single-neck flask, and then add in the methyl alcohol/ammonia solution just prepared in the second paragraph. Thereafter, place a suitable sized balloon over the flask, and secure the balloon to the flask using a metal ring clamp. Then heat the contents in the flask to about 40 Celsius for about 4 days. Note: the balloon will inflate and deflate sporadically during the heating process. The balloon is deigned to keep the contents of the flask under pressure to properly carryout the reaction. If during the heating process, the balloon pops or explodes, quickly replace with another one, and continue the operation for the necessary amount of remaining time. *Note: the pressure process just described, whereby a balloon is placed over a flask, can be substituted by using a conventional steel pipe with threads at both ends. To carryout the steel pipe technique, pour all necessary materials (as described for the balloon technique), into a thick walled stainless steel pipe, and then seal both ends with the corresponding steel caps. The threads at each end should be wrapped with Teflon tape prior to screwing in the end caps. Then place the entire pipe, and submerge it into a water bath and heat at the desired temperature for the desired time. Note: this process can be dangerous and can lead to pressure explosions. Carryout the process in an area that can contain any such explosion, and maintain a safe distance away during the operation—just to be on the safe side.* After the heating process, remove the heat source, and allow the reaction mixture to cool to room temperature. Note: monitor the balloon so it does not get sucked into the flask due to backpressure. Thereafter, remove the contents of the flask, or steel pipe, and place them into a suitable distillation apparatus, and distill-off the methyl alcohol and any excess ammonia by distillation at 68 Celsius. When no more methyl alcohol passes over or is collected, stop the distillation process, and recover the left over remaining residue (after it has cooled). Then place this left over collected residue into a suitable beaker, and then add in 100 milliliters of a 10% sodium hydroxide solution, and then stir the entire mixture for about 30 minutes. Thereafter, extract this entire mixture with three 50-milliliter portions of methylene chloride, and after the extraction process, combine all methylene chloride portions (if not already done so), and then dry this combined methylene chloride portion by adding to it, 15 grams of anhydrous magnesium sulfate. Note: after each extraction, the methylene chloride will be the lower layer each time. After adding in the sodium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Then place this filtered methylene chloride portion into a distillation apparatus, and remove the methylene chloride at 40 Celsius by distillation. When no more methylene chloride passes over or is collected, remove the left over remaining residue (after it has cooled), and then dissolve this left over residue into 100 milliliters of dry diethyl ether. Last but not least, place this ether mixture into an ice bath, and chill to 0 Celsius. Thereafter, bubble into this ether mixture, 15 grams of hydrogen chloride gas (excess), and after the addition, allow the acidified ether mixture to stand for 1 hour at 0 Celsius. Then filter-off the precipitated MMDA product, wash with two 25-millilter portions of diethyl ether, and then vacuum dry or air-dry the crystals.

Note: As mentioned many times in this book, other salts of the freebase compound can be obtained by treating the freebase solution with the desired acid. Other salts of the freebase MMDA such as the sulfate, tartrate, citrate, and phosphate can be prepared by adding the corresponding acid to the ether mixture of the freebase MMDA compound obtained at the end of the above paragraph. For sulfuric acid or tartaric acid, the usual mole quantity should be added to the usual mole quantity of the freebase MMDA which is dissolved in the ether. Note: as with all of these notes everywhere in this book, this means 1 mole of acid per 2 moles of the MMDA on a dry bases—not the total weight of the ether mixture. For citric acid or phosphoric acid, the usual mole quantity (1 mole) of citric acid or phosphoric acid should be added to the usual mole quantity of the freebase MMDA (3 moles) in the ether mixture. The ether mixture before treatment with any of the aforementioned acids, should be chilled in an ice bath at 0 Celsius prior to addition. After treatment with the corresponding acid in each of these cases, the ether mixture can be filtered to recover the precipitated crystals. The crystals should then be vacuum dried or air-dried. As expected, all salts of MMDA have similar properties. Note: the citrate salt may be more potent then the hydrochloride.

0023. BOH. beta-Methoxy-3,4-methylenedioxyphenethylamine hydrochloride. *2-(1,3-benzodioxol-5-yl)-2-methoxyethanamine*



Structure | 3D model

BOH forms brilliant white crystals with a melting point of 152 Celsius. It is capable of forming a monohydrate, with a melting point of 106 Celsius. BOH is a mild psychedelic amphetamine with peculiar properties. Its effects are rarely felt with one single dose, and the best results from its use are encountered when it is taken in several smaller doses over a specified time period. Effects of the drug when taken in one single large dose include, general bodily discomfort, meaning nausea, headache, bowel discomfort, stomach problems, and cold chills; however, when the drug is taken in 20 to 40 milligram doses at a time, and several times over a period of several hours, the effects produced include, an increase in motivation, mental awareness, mood enhancements, and overall good feelings of well being. The exact dose for maximum effect should be the following: take 20 to 40 milligram doses once per hour for 3 to 4 hours. BOH is not your normal psychedelic amphetamine, but it is capable of producing mild hallucinogens, but only in the areas of mood, and feelings—meaning, the drug usually produces enhancements of mood and feelings, rather then sight, sound, touch, ect., ect., Early research on this drug proclaimed it to be a "prodrug", meaning it is capable of metabolizing in the body over unknown time periods to produce one or more active drugs with more effects upon the body then the original drug—this may be the basis for the 20 to 40 milligram dose per hour.

This substance is a controlled substance (psychedelic amphetamine) as listed in the US code of Federal regulations.

Toxicity: High	**Rate of onset (average):** Moderate (may take up to 45 minutes for effects to be realized)
Stimulation dosage (ingestion): 20 to 40 milligrams once every hour for 3 to 4 hours	**Duration of effects (average):** 6 hours per trip
Stimulation dosage (inhalation): none	**Habit forming potential:** Low
Stimulation dosage (injection): unknown	**Estimated value U.S. (based on procedure):** $29 per gram

Procedure A: Preparation of BOH

Materials:

1. 15 grams of piperonal (see intermediate-0011, procedure B, of step 3, and intermeidate-0024 for its preparation)	11. 1.2 milliliters of 98% sulfuric acid
2. 85 milliliters of glacial acetic acid.	12. 65 milliliters of 99% isopropyl alcohol
3. 10 milliliters of nitroethane	13. 150 milliliters of a 15% sodium hydroxide solution
4. 5 milliliters of cyclohexylamine	14. 300 milliliters of a 10% sulfuric acid solution
5. 325 milliliters of methyl alcohol	15. 30 grams of sodium hydroxide
6. 2.7 grams of metallic sodium	16. 15 grams of anhydrous magnesium sulfate
7. 320 milliliters of methylene chloride	17. 15 grams of 35 to 38% hydrochloric acid
8. 250 milliliters of a 5% sodium bicarbonate solution	18. 50 milliliters of diethyl ether

9. 6.8 grams of lithium aluminum hydride	19. 50 milliliters of toluene
10. 250 milliliters of tetrahydrofuran (THF)	

Summary: BOH is prepared in a two-step process starting with the formation of 1-(3,4-methylenedioxyphenyl)-2-nitroethane. This intermediate is prepared by treating 3,4-methylenedioxy-beta-nitrostyrene with sodium methoxide in methyl alcohol. The 3,4-methylenedioxy-beta-nitrostyrene is prepared by condensing piperonal with nitroethane in the usual manner. After the initial reactions, the desired 1-(3,4-methylenedioxyphenyl)-2-nitroethane is then collected by methylene chloride extraction, followed by removal of the solvent, and then recrystallization from methyl alcohol. The collected 1-(3,4-methylenedioxyphenyl)-2-nitroethane is then converted into BOH by reduction with lithium aluminum hydride in tetrahydrofuran. The reaction is rather mild, and afterwards, the reaction mixture is treated with alcohol and sodium hydroxide to neutralize any excess lithium aluminum hydride, and the resulting reaction mixture is then filtered. The filtered reaction mixture is then evaporated, and the left over residue is then acidified with sulfuric acid. The acidified mixture is then extracted with methylene chloride to remove impurities, and the resulting extracted mixture is then treated with base to liberate the freebase BOH, which is then extracted into methylene chloride. The methylene chloride is then stripped, and the left over residue is then dissolved in alcohol, treated with acid, and then finally precipitated with ether.

Hazards: Extinguish all flames before using cyclohexylamine, methyl alcohol, isopropyl alcohol, and toluene. Extinguish all flames before using tetrahydrofuran, diethyl ether, and nitroethane, all three of which are highly flammable, and capable of forming explosive mixtures with air—use caution. Wear gloves when handling glacial acetic acid, sodium hydroxide, sulfuric acid and hydrochloric acid, all of which are capable of forming skin irritation. Wear gloves when handling metallic sodium and lithium aluminum hydride, both of which are very reactive and dangerous in contact with water and other chemicals—use caution.

Procedure:

Personnel notes for procedure A: BOH

Step 1: Preparation of 1-(3,4-methylenedioxyphenyl)-2-nitroethane

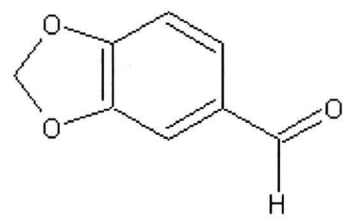
Into a suitable reflux apparatus, place 15 grams of piperonal (see intermediate-0011, procedure B, of step 3, and intermeidate-0024 for its preparation), followed by 50 milliliters of glacial acetic acid. Thereafter, add in 10 milliliters of nitroethane, followed immediately by 5 milliliters of cyclohexylamine as catalyst. Then reflux the entire mixture for 1 hour at 100 Celsius. After refluxing for 1 hour, stop the reflux process, and then allow the reaction mixture to cool to room temperature. Thereafter, place the reaction mixture into an ice bath, and chill to 0 Celsius for about 1 hour. Then filter-off the precipitated crystals, wash the crystals with 10 milliliters of glacial acetic acid, and then vacuum dry or air-dry the crystals. Thereafter, place (suspend) the dried crystals into 50 milliliters of warm methyl alcohol (pre-heated to about 40 Celsius), and then stir the entire mixture for about 30 minutes. Thereafter, filter-off the insoluble crystals, and then vacuum dry or air-dry the crystals. The result will be about 12 grams of 3,4-methylenedioxy-beta-nitrostyrene. Now, into a suitable beaker or flask (equipped with stirring means, and thermometer), add 10 grams of the 3,4-methylenedioxy-beta-nitrostyrene (just prepared), followed by 50 milliliters of methyl alcohol. Then prepare a sodium methoxide solution by slowly adding and dissolving 2.7 grams of metallic sodium into 50 milliliters of methyl alcohol. Note: before adding the metallic sodium to the methyl alcohol, place the methyl alcohol into an ice bath, and chill to 0 Celsius. During the addition of the metallic sodium, rapidly stir the methyl alcohol, and keep its temperature below 10 Celsius. After this sodium methoxide solution has been prepared, add this sodium methoxide solution to the 3,4-methylenedioxy-beta-nitrostyrene/methyl alcohol mixture over a period of about 5 minutes. During the addition of the sodium methoxide solution, rapidly stir the 3,4-methylenedioxy-beta-nitrostyrene/methyl alcohol mixture. After adding the sodium methoxide solution, immediately add in 25 milliliters of glacial acetic acid, and shortly after this addition, add in 150 milliliters of water, and then rapidly stir the reaction mixture for about 10 minutes. After 10 minutes, stop stirring, and allow the reaction mixture to stand for several minutes. Thereafter, extract this two-phase reaction mixture with one 100-milliliter portion of methylene chloride. After the extraction, briefly wash this single methylene chloride portion with 250 milliliters of a 5% sodium bicarbonate solution, and thereafter, wash the single methylene chloride portion with 250 milliliters of water. Note: during the extraction and washing portions, the methylene chloride will be the lower layer each time. Now, place the recovered methylene chloride portion into a distillation apparatus, and distil-off the methylene chloride at 40 Celsius. When no more methylene chloride passes over or is collected, stop the distillation process, and recover the left over remaining oily reside (after it has cooled). Finally, recrystallize this left over residue from 100 milliliters of methyl alcohol, and after the

recrystallization process, vacuum dry or air-dry the collected crystals. Note: a second recrystallization from 75 milliliters of methyl alcohol would be recommend.

Step 2: Preparation of BOH

Into a suitable reflux apparatus (equipped with stirring means, and thermometer), place 6.8 grams of lithium aluminum hydride, followed by 200 milliliters of tetrahydrofuran (THF). Thereafter, slowly stir (to prevent bumping) the mixture to dissolve the tetrahydrofuran. Thereafter, place this mixture into an ice bath, and chill to about 0 Celsius. When the mixtures temperature reaches 0 Celsius, slowly add in, 1.2 milliliters of 98% sulfuric acid, and then rapidly stir the mixture for about 10 minutes. Thereafter, add in 6 grams of the product obtained in step 1, over a period of about 1 minute. During the addition, rapidly stir the reaction mixture and maintain its temperature below 5 Celsius. After the addition, continue to stir the reaction mixture for about 5 minutes, and then remove the ice bath. Afterwards, reflux the entire reaction mixture at 66 Celsius for a short period of time. Note: this short period of time is determined by the gas evolution that will take place during the heating process. When the gas evolution becomes rather vigorous, remove the heat source, and allow the reaction mixture to cool to room temperature. Thereafter, wait for the gas evolution to slow down, and then reflux the reaction mixture again at 66 Celsius for about 10 to 15 minutes. Thereafter, remove the heat source, and allow the reaction mixture to cool to room temperature. Then pour the entire reaction mixture into a suitable sized beaker, and then add in 50 milliliters of 99% isopropyl alcohol, followed by 150 milliliters of a 15% sodium hydroxide solution. After the addition of the alcohol and sodium hydroxide, rapidly stir the entire reaction mixture for about 10 minutes. Thereafter, filter-off any insoluble materials, and then quickly wash these filtered-off materials with two 25-milliliter portions of tetrahydrofuran. Then combine these two tetrahydrofuran portions with the filtered reaction mixture. Now, place the combined reaction mixture into a distillation apparatus, and distill-off the tetrahydrofuran at 66 Celsius. When no more tetrahydrofuran passes over or is collected, stop the distillation process, and recover the left over remaining oily residue (after it has cooled), and then carefully and slowly dissolve this oily residue into 300 milliliters of a 10% sulfuric acid solution. Then quickly extract this acidic mixture with three 40-millilter portions of methylene chloride, and after the extraction process, combine all methylene chloride portions (if not already done so), and then discard or recycle the methylene chloride portions. Note: after each extraction, the methylene chloride will be the upper layer each time. Now, after the extraction process, place the recovered lower acidic layer into a suitable sized beaker, and then add in a sodium hydroxide solution prepared by adding and dissolving 30 grams of sodium hydroxide into 150 milliliters of water. Note: sodium hydroxide generates excessive heat when dissolved in water, so allow the solution to cool to room temperature before using. After the addition of the sodium hydroxide solution, rapidly stir the now alkaline mixture for about 30 minutes. Then extract the entire alkaline mixture with two 50-milliliter portions of methylene chloride, and after the extraction process, combine all methylene chloride portions (if not already done so), and then dry this combined methylene chloride portion, by adding to it, 15 grams of anhydrous magnesium sulfate. Then stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Note: after each extraction, the methylene chloride will be the lower layer each time. Then place this filtered methylene chloride portion into a distillation apparatus, and distill-off the methylene chloride at 40 Celsius. When no more methylene chloride passes over or is collected, remove the heat source, and collect the left over remaining residue (after it has cooled). Thereafter, dissolve this collected residue into 15 milliliters of 99% isopropyl alcohol, and then stir the mixture for about 10 minute. Thereafter, filter the alcohol mixture to remove any insoluble materials. Finally, add to this alcohol mixture, 15 grams of 35 to 38% hydrochloric acid (muritatic acid of 31% will work), and after the addition of the hydrochloric acid, add in 25 milliliters of diethyl ether, and then briefly stir the mixture for about 10 minutes. Finally, filter-off the precipitated crystals of the desired product, and then vacuum dry or air-dry them. Now, place (suspend) the dried product into 50 milliliters of toluene, and then heat at 100 Celsius for about 30 minutes. Note: during this heating process, rapidly stir the toluene mixture. Thereafter, remove the heat source, and allow the toluene mixture to cool. Then filter-off the crystals of the BOH, wash with two portions of 25 milliliters each of diethyl ether, and then vacuum dry or air-dry the crystals.

Intermediate-0024. Piperonal. *1,3-benzodioxole-5-carbaldehyde*



Piperonal is a major starting point for the preparation of numerous psychedelic amphetamines and related compounds. Piperonal itself is not a controlled substance, but persons producing it should be warned as it is listed by the DEA as a "suspect" compound, meaning its production, distribution, and sale are monitored by the drug enforcement agencies. Note: Piperonal can also be made from black pepper as described in Intermediate-0011. Piperonylacetone, procedure B.

Procedure A: Preparation of piperonal from protocatechualdehyde via vanillin

Materials:

1. 12 grams of vanillin (see intermediate-0019. 3,4,5-TMB, procedure B, step 1)	12. or 450 milliliters of diethyl ether
2. 128 milliliters of dry methylene chloride	13. or 450 milliliters of a 5% sodium hydroxide solution
3. 11.6 grams of anhydrous aluminum chloride	14. or 150 milliliters of a 10% sulfuric acid solution
4. 27 grams of pyridine	15. or 10 grams of anhydrous sodium sulfate
5. 150 milliliters of a 10% hydrochloric acid solution	16. 60 milliliters of dimethylsulfoxide (DMSO)
6. 475 milliliters of diethyl ether	17. 9.3 grams of sodium hydroxide
7. 15 grams of anhydrous sodium sulfate	18. 10 grams of anhydrous sodium sulfate
8. or 193 milliliters of nitrobenzene	19. or 50 milliliters of methylene chloride
9. or 12.6 grams of vanillin (see intermediate-0019. 3,4,5-TMB, procedure B, step 1)	20. or 1.2 grams of tetrabutylammonium bromide
10. or 44 grams of anhydrous aluminum bromide	21. or 5 grams of sodium hydroxide
11. or 830 milliliters of a 5% hydrochloric acid solution	22. or 50 milliliters of diethyl ether

Summary: Piperonal is prepared in a two-step process starting with the formation of protocatechualdehyde. This intermediate is very important and can be made by refluxing vanillin with pyridine and aluminum chloride in methylene chloride. The reaction is rather general, and afterwards, the reaction mixture is treated with dilute acid, and the resulting two-phase mixture is then separated, and the lower aqueous layer is then extracted with ether. The ether is then removed in the usual manner, and the remaining residue is then recrystallized from ether. The resulting product is the desired intermediate protocatechualdehyde. Protocatechualdehyde can also be made from vanillin in a modified process by refluxing vanillin with nitrobenzene in the presence of anhydrous aluminum bromide. After the reflux period, the reaction mixture is drowned into dilute acid, extracted with ether, treated with base, and then finally acidified. The acidified mixture is then extracted with ether, and the ether extract is then removed to recover the desired product of protocatechualdehyde. The protocatechualdehyde is then converted into the desired piperonal by reaction with methylene chloride in the presence of sodium hydroxide under high temperature. The reaction is mild, and afterwards, the desired piperonal is collected by steam distillation, followed by extraction into ether. The ether extract is then evaporated, and the remaining residue is then recrystallized from ether to afford the desired product of piperonal. In a modified process, the protocatechualdehyde is converted into piperonal by heating with excess methylene chloride in the presence of sodium hydroxide under heat and pressure. After the reaction, the reaction mixture is separated into two layers, and the methylene chloride layer is then evaporated to recover the dissolved product, which is purified by recrystallization from ether.

Hazards: Use proper ventilation when handling diethyl ether, which is highly flammable, and capable of forming explosives mixtures with air—use great care. Use extreme caution when handling nitrobenzene, which is highly toxic and can be absorbed through the skin. Avoid eye, skin, nose, and throat contact at all cost. Wear gloves when handling hydrochloric acid, sulfuric acid, and sodium hydroxide, all of which are capable of causing skin irritation. Anhydrous aluminum chloride should be handled with care as it is capable of reacting violently with water—this applies to anhydrous aluminum bromide as well. Wear gloves and use proper ventilation when handling dimethylsulfoxide, which is toxic and can be absorbed through the skin.

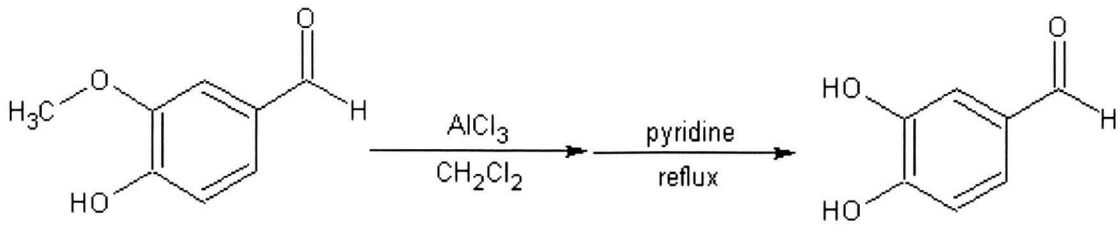
Procedure:

Personnel notes for procedure A: Piperonal

Step 1: Preparation of protocatechualdehyde from vanillin (method 1)

Into a suitable reflux apparatus, fitted with reflux condenser (fitted with calcium chloride drying tube to exclude moisture), motorized stirrer and thermometer, place 12 grams of vanillin (see intermediate-0019. 3,4,5-TMB, procedure B, step 1), followed by 120 milliliters of dry methylene chloride. Thereafter, briefly stir the entire mixture to form a uniform mixture. Then quickly add in, 11.6 grams of anhydrous aluminum chloride (add through the top of the reflux condenser—simply remove the calcium chloride drying tube, and then quickly re-attach it after adding in the aluminum chloride). After adding in the aluminum chloride, place the entire reaction mixture into a suitable sized cold-water bath, and then slowly add in, 27 grams

of pyridine, in small portions at a time at such a rate as to keep the reaction mixtures temperature below 38 celsius at all times. During the addition of the pyridine, rapidly stir the reaction mixture and maintain its temperature below 35 Celsius at all times. Note: the pyridine can be added through the top of the reflux condenser in the same manner as the aluminum chloride. After the addition of the pyridine, reflux the entire reaction mixture at 45 to 50 Celsius for about 28 hours with constant stirring. After refluxing for about 28 hours, remove the heat source and allow the reaction mixture to cool to room temperature. Thereafter, pour the entire reaction mixture into a suitable sized beaker, and then add in 150 milliliters of a 10% hydrochloric acid solution, and then vigorously stir the entire two-phase mixture thereafter for about 30 minutes. Then place this entire two-phase mixture into a seperatory funnel, and remove the lower aqueous layer. Note: the upper methylene chloride layer can be recycled or discarded if desired. Now to the lower recovered aqueous layer, extract this aqueous layer with three 50-millilter portions of diethyl ether, and after the extraction process, combine all ether portions (if not already done so), and then dry this combined ether portion by adding to it, 15 grams of anhydrous sodium sulfate. Note: after each extraction portion, the ether will be the upper layer each time. After the addition of the sodium sulfate, stir the entire ether mixture for about 10 minutes, and then filter-off the sodium sulfate. Finally, place the filtered ether portion into a distillation apparatus, and distill-off the ether at 40 Celsius. When no more ether passes over or is collected, stop the distillation process, and then recover the left over remaining residue (after it has cooled), and then recrystallize this recovered residue from 100 milliliters of fresh diethyl ether. After the recrystallization process, vacuum dry or air-dry the collected pale yellow crystals. The result should be about 9 grams of the desired product with a melting point of 154 Celsius.

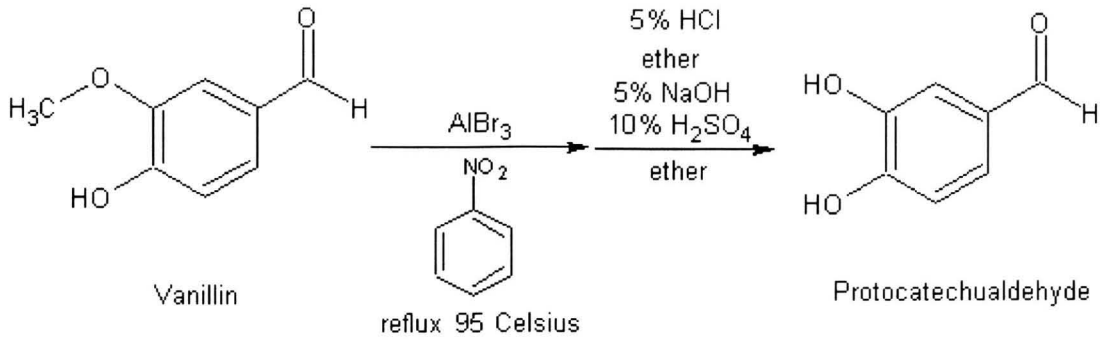


Vanillin

Protocatechualdehyde

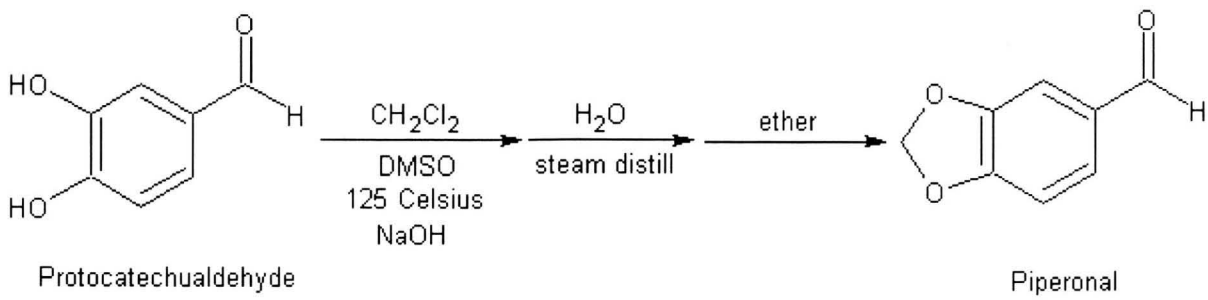
Step 1: Preparation of protocatechualdehyde from vanillin (method 2)

Into a suitable beaker (equipped with motorized stirrer or magnetic sitter, and thermometer), add 38 milliliters of nitrobenzene followed by 12.6 grams of vanillin (see intermediate-0019. 3,4,5-TMB, procedure B of step 1). Thereafter, stir the mixture to form a uniform mixture. Then prepare a second solution by adding 50 milliliters of nitrobenzene into a clean suitable sized beaker, and then add in 44 grams of anhydrous aluminum bromide. Then stir this mixture to form a uniform mixture. Thereafter, place the nitrobenzene/vanillin mixture into a cold-water bath, and chill to about 15 Celsius. Thereafter, add in, the nitrobenzene/aluminum bromide mixture while rapidly stirring the nitrobenzene/vanillin mixture. After the addition, stir the entire gelled mixture for about 10 minutes, and then add in 105 milliliters of fresh nitrobenzene, and then stir the entire mixture for about 30 minutes. Then place this entire mixture into a suitable sized reflux apparatus, and reflux the entire mixture at 95 Celsius for about 30 minutes. After refluxing for about 30 minutes, remove the heat source, and allow the mixture to cool to room temperature. Thereafter, pour the entire mixture into a suitable sized beaker, and then add in 830 milliliters of a 5% hydrochloric acid solution. After the addition, rapidly stir the acidic mixture for about 1 hour. Then extract the entire acidic mixture with three 50-milliliter portions of diethyl ether, and after the extraction process, combine all ether portions (if not already done so). Note: after each extraction process, the ether will be the upper layer each time. Note: the lower aqueous layer containing the nitrobenzene can be recycled. Now, to this combined ether portion, extract this combined ether portion with three 150-milliiilter portions of a 5% sodium hydroxide solution. Note: after each alkaline extraction, the sodium hydroxide portion will be the lower layer each time—the upper ether layer can be recycled or discarded if desired after each extraction. After the extraction, combine all lower aqueous sodium hydroxide portions (if not already done so), and thereafter, wash this combined aqueous sodium hydroxide portion with two 25-milliliter portions of diethyl ether. Note: after each washing portion, the aqueous sodium hydroxide portion will be the lower layer each time—the upper ether portion can be recycled after each washing. After the washing portion, place this aqueous sodium hydroxide layer into a suitable sized beaker, and then add in 150 milliliters of a 10% sulfuric acid solution. After the addition of the sulfuric acid, rapidly stir the entire mixture for about 30 minutes. Finally, extract the entire acidic mixture with three 50-milliliter portions of diethyl ether, and after the extraction process, combine all ether portions (if not already done so), and then dry this combined ether portion by adding to it, 10 grams of anhydrous sodium sulfate. Note: after each extraction process, the ether will be the upper layer each time. After the addition of the sodium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the sodium sulfate. Then place this filtered ether portion into a distillation apparatus, and distill-off the ether at 40 Celsius. When no more ether passes over or is collected, stop the distillation process, and recover the left over remaining residue (after it has cooled). Thereafter, recrystallize this left over recovered residue from 100 milliliters of diethyl ether, and after the recrystallization process, vacuum dry or air-dry the filtered-off crystals. The result will be about 10 grams of the desired product.



Step 2: Preparation of piperonal (method 1)

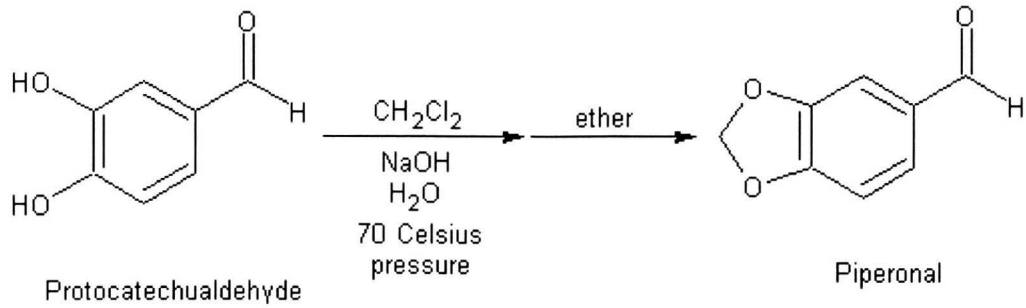
Into a suitable reflux apparatus (equipped with thermometer and motorized stirrer), place 15 milliliters of methylene chloride, followed by 60 milliliters of dimethylsulfoxide (DMSO). Thereafter, carefully heat the entire solvent mixture to about 125 Celsius, and begin a reflux. When the solvent begins to reflux, add in small portions (through the top of the reflux condenser), a dry solid mixture prepared by thoroughly mixing together, 13 grams of protocatechualdehyde (obtained in step 1) and 9 grams of sodium hydroxide. Note: each portion should be about 2 grams and should be added once every 5 minutes. During the addition of the solid mixture, rapidly stir the methylene chloride/DMSO solvent mixture and maintain its temperature around 125 Celsius. After the addition of the solid mixture, continue to reflux and stir the reaction mixture at 125 Celsius for about 15 additional minutes, and thereafter, add in, in one portion, 3 milliliters of methylene chloride, followed by 300 milligrams of sodium hydroxide. Then continue to reflux the reaction mixture and rapidly stir it for about 1 hour. After 1 hour, remove the heat source, and allow the reaction mixture to briefly cool to room temperature. Then pour the entire reaction mixture into a 3-neck flask, fitted with an addition funnel, condenser (fitted with receiver flask), and motorized stirrer or other stirring means. Then add to the reaction mixture in the 3-neck flask, 60 milliliters of water. Then pour 250 milliliters of water into the addition funnel, and then steam distill the reaction mixture (in the 3-neck flask) at 100 Celsius. Note: the exact time needed to properly carryout the steam distillation may vary; however, when the contents in the receiver flask reach about 100 milliliters, stop the steam distillation process. Note: during the entire steam distillation process, small increments of water (20 to 50 milliliters at a time) should be added from the addition funnel to maintain a proper water level to carryout the steam distillation process. When about 100 milliliters of liquid has been collected in the receiver flask, stop the steam distillation process, and recover the liquid contents in the receiver flask, by pouring them into a suitable sized beaker. Then extract this entire liquid with three 50-millilter portions of diethyl ether, and after the extraction process, combine all ether portions (if not already done so), and then dry this combined ether portion, by adding to it, 10 grams of anhydrous sodium sulfate. Note: after each extraction portion, the ether will be the upper layer each time. After adding in the sodium sulfate, stir the entire ether portion for about 10 minutes (to absorb moisture), and then filter-off the sodium sulfate. Finally, place the filtered ether mixture into a distillation apparatus, and distill-off the ether at 40 Celsius. When no more ether passes over or is collected, stop the distillation process, and recover the left over remaining residue (after it has cooled). Then recrystallize this left over remaining residue from 75 milliliters of fresh diethyl ether, and after the recrystallization process, vacuum dry or air-dry the recovered crystals of piperonal.



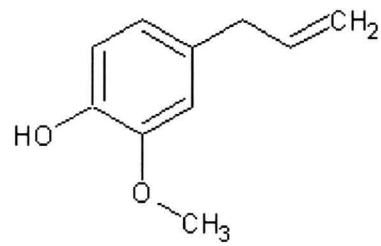
Step 2: Preparation of piperonal (modified method 2)

Into a suitable single neck flask, place 50 milliliters of methylene chloride, followed by 1.2 grams of tetrabutylammonium bromide as catalyst. Thereafter, prepare a mixture by first, preparing a sodium hydroxide solution prepared by adding and dissolving 5 grams of sodium hydroxide into 7 milliliters of water. Note: sodium hydroxide generates excessive heat when dissolved in water, so allow the alkaline solution to cool before continuing. Thereafter, thoroughly blend this sodium hydroxide solution with 5.5 grams of protocatechualdehyde (obtained in step 1). Thereafter, add only 3 grams of this total mixture to the methylene chloride and catalyst mixture, and then place a suitable sized balloon over the single neck flask, and secure the balloon to the flask using a metal ring clamp. Then heat the contents in the flask to about 70 Celsius for about 15 minutes.

After 15 minutes, quickly remove the balloon once again, and then add in another 3-gram portion of the protocatechualdehyde/sodium hydroxide mixture, and then re-attach the balloon and then continue heating at 70 Celsius for 15 minutes. After the 15-minute period, repeat this process 3 more times with three 3-gram potions of the protocatechualdehyde/sodium hydroxide mixture. Note: the third and final portion will not weigh 3 grams. After the third and final addition, and after heating for the desired 15 minutes, continue heating the reaction mixture at 70 Celsius for an additional 20 minutes. Note: during the heating process the balloon will inflate and deflate sporadically during the heating process. The balloon is deigned to keep the contents of the flask under pressure to properly carryout the reaction. If during the heating process, the balloon pops or explodes, quickly replace with another one, and continue the operation for the necessary amount of remaining time. After the heating process, remove the heat source, and allow the reaction mixture to cool to room temperature. Note: monitor the balloon as the balloon cools to prevent it from getting sucked into the flask due to backpressure. Then pour the entire reaction mixture into a seperatory funnel, and then recover the lower methylene chloride layer. Thereafter, place this methylene chloride layer into a distillation apparatus, and distill-off the methylene chloride. When no more methylene chloride passes over or is collected, stop the distillation process, and recover the left over remaining residue (after it has cooled). Then recrystallize this left over remaining residue from 50 milliliters of diethyl ether, and after the recrystallization process, vacuum dry or air-dry the crystals of piperonal.



Intermediate-0025. Eugenol. *4-allyl-2-methoxyphenol*



Eugenol forms a colorless to pale yellowish liquid with a boiling point of 255 Celsius. Eugenol slowly turns dark on exposure to air, so it should be stored in airtight bottles in a cool place. Eugenol has a powerful odor of cloves, from which it is readily extracted from ordinary spice cloves. Eugenol has a melting point of –9 Celsius, so the oil may crystallize on standing under cold temperatures. Eugenol is miscible with alcohol, methylene chloride, and ether, but insoluble in water. Eugenol is a major starting point for the preparation of psychedelic amphetamines.

Procedure A: Extraction of eugenol from cloves

Materials:

1. 100 grams of cloves (regular store bought cloves) found in any grocery store under the spice section	4. 300 milliliters of a 5% hydrochloric acid solution
2. 360 milliliters of methylene chloride	5. 50 milliliters of a 23% sodium chloride solution
3. 300 milliliters of a 5% potassium hydroxide solution	6. 15 grams of anhydrous sodium sulfate

Summary: Eugenol can easily be extracted from cloves in a series of simple steps starting with steam distillation. The cloves are steam distilled to free the eugenol, which gets carried over as an oil with the water. The oil/water mixture is then extracted with methylene chloride, and the resulting methylene chloride/eugenol extract is then extracted with a dilute potassium hydroxide solution. During this alkaline extraction, the desired eugenol is taken up into the aqueous potassium hydroxide solution. After separation of the eugenol from the methylene chloride and impurities, the eugenol is then treated with acid, whereupon it is then extracted into fresh methylene chloride. The methylene chloride is then evaporated to recover the essential eugenol in 98% purity.

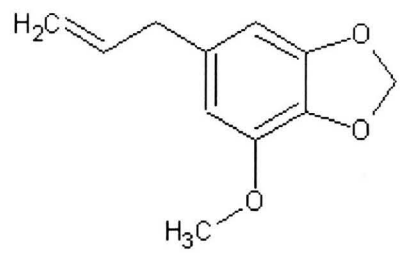
Hazards: Wear gloves when handling potassium hydroxide and hydrochloric acid, both of which are capable of causing skin burns.

Procedure:

Personnel notes for procedure A: Eugenol

Into a suitable steam distillation apparatus (fitted with a 250 milliliter addition funnel, or better), place 100 grams of cloves (regular store bought cloves). Thereafter, add in 500 milliliters of water, and then add 250 milliliters of water to the addition funnel. This 250-milliliter addition funnel should contain about 200 milliliters of water at all times, and the water therein should be added to the cloves and water mixture periodically to keep the flasks water volume at around 500 milliliters all throughout the steam distillation process. Then heat the cloves and water mixture to 105 to 110 Celsius, and allow the mixture to be steam distilled. The process should take about 150 minutes, and thereafter, stop the steam distillation process, and then recover the oily distillate in the receiver flask. Then extract this oily distillate with three 50-millilter portions of methylene chloride, and after the extraction, combine both methylene chloride portions (if not already done so). Note: after each extraction, the methylene chloride will be the bottom layer each time. After the extraction, the upper water layer can be discarded. Now, extract the combined methylene chloride portion with six 50-milliliter portions of a 5% potassium hydroxide solution. After the extraction, combine all aqueous alkaline portions (if not already done so), and then briefly wash this combined aqueous alkaline portion with one portion of 50 milliliters of methylene chloride. Note: after the extraction and washing, the aqueous alkaline portion will be the upper layer each time. After the extraction and washing, the methylene chloride can be recycled if desired. Then place this combined aqueous alkaline portion into a large beaker, and then carefully add in, slowly, 250 to 300 milliliters of a 5% hydrochloric solution. Note: more or less acid may or may not be needed, and the acid is added soley to bring the pH of the aqueous mixture (in the beaker) to about 1—add as much acid as needed to reach a pH of about 1. After adding the acid, moderately stir the entire acidic mixture for about 30 minutes. Then, extract this entire acidic mixture with four 40-milliliter portions of methylene chloride. After the extraction process, combine all methylene chloride portions (if not already done so), and then wash this combined methylene chloride portion with one 50-milliilter portion of water, followed by one 50 milliliter portion of a 23% sodium chloride solution. Note: after the extraction and washings, the methylene chloride will be the lower layer each time. After the extraction and washing portions, dry the washed methylene chloride portion by adding to it, 15 grams of anhydrous sodium sulfate, and then stir the entire mixture for about 10 minutes—thereafter, filter-off the sodium sulfate. Finally, place this filtered dried methylene chloride portion into a distillation apparatus, and distill-off the methylene chloride at 40 Celsius. When no more methylene chloride passes over or is collected, remove the left over remaining pale yellow oil (after it has cooled), and then store it in an amber glass bottle in a refrigerator until use. Note: the eugenol at this point will have a purity of about 98%.

Intermediate-0026. Myristicin. *6-allyl-4-methoxy-1,3-benzodioxole*



Myristicin forms a colorless to yellowish oil (depending on purity), with a boiling point (at 40 milliliters of mercury) of 173 Celsius. Myristicin exits naturally in nutmeg, carrots, and parsley, from which it can be extracted—especially from the corresponding oils. Myristicin is used in the preparation of a few psychedelic amphetamines, mainly MMDA.

Procedure A: Preparation of myristicin from eugenol

Materials:

1. 12 milliliters of eugenol (see intermediate-0025. Eugenol)	13. 12 milliliters of a 6% hydrogen peroxide solution
2. 90 milliliters of glacial acetic acid	14. 50 milliliters of a pre-chilled ice-cold 10% hydrochloric

	acid solution
3. 48 grams of hexamine	15. 50 milliliters of a 5% sodium bicarbonate solution
4. 5 grams of steel wool	16. 50 grams of regular sodium chloride (table salt)
5. 60 milliliters of 35 to 38% hydrochloric acid	17. 50 milliliters of ice cold 23% sodium chloride solution
6. 410 milliliters of diethyl ether	18. 50 milliliters of 10% hydrochloric acid
7. 40 grams of sodium hydroxide	19. 20 grams of anhydrous sodium sulfate
8. 100 milliliters of a 20% sodium hydroxide solution	20. 240 milliliters of dry acetone
9. 90 milliliters of a 20% hydrochloric acid solution	21. 9 milliliters of methylene bromide
10. 75 milliliters of petroleum ether	22. 36 grams of potassium carbonate
11. 20 milliliters of pyridine	23. 100 milliliters of a 10% sodium carbonate solution
12. 23 milliliters of a 10% sodium hydroxide solution	

Summary: Myristicin can be prepared in a three-step process starting with the formation of eugenol aldehyde. This aldehyde intermediate is best prepared by reacting eugenol with hexamine in the presence of glacial acetic acid and steel wool. Note: the steel wool may be replaced with other metal wools such as copper, zinc, tin, nickel, ect., ect., and better yields may result. The reaction mixture is then refluxed, and then treated with hydrochloric acid. After the acid treatment, the desired product is collected by an exhaustive extraction process, whereby the product is extracted into ether, treated with sodium hydroxide, and converted into the insoluble sodium salt of eugenol aldehyde. The sodium salt is then collected by filtration, dissolved into water, and then broken down by the addition of acid. The acid addition allows for the precipitation of the sodium free compound, which separates out as pale crystals. The pale crystals are then recrystallized from a suitable solvent. The refined crystals of eugenol aldehyde are then converted into hydroxy eugenol by reaction with hydrogen peroxide in the presence of sodium hydroxide and pyridine. The reaction is relatively simple, and afterwards, the reaction mixture is extracted, and the combined solvent extracts are then evaporated to leave behind an oil, composed predominantly of the desired hydroxy eugenol. The hydroxy eugenol is converted into myristicin by reaction with methylene bromide in the presence of potassium carbonate and acetone. The reaction mixture is refluxed, and the resulting mixture is then extracted with solvent. The combined solvent extract is then removed in the usual manner to afford the desired myristicin product.

Hazards: Extinguish all flames before using diethyl ether, petroleum ether, and acetone as all three are highly flammable and capable of volatizing quite readily—especially ether. Ether is capable of forming explosive mixtures with air so use caution. Wear gloves when handling hydrochloric acid, and sodium hydroxide, as they are capable of producing skin irritation.

Procedure:

Personnel notes for procedure A: Myristicin

Step 1: Preparation of eugenol aldehyde

Into a suitable reflux apparatus, fitted with motorized stirrer or other stirring means and empty addition funnel, place 12 milliliters of eugenol, followed by 90 milliliters of glacial acetic acid. Thereafter, rapidly blend the mixture to form a uniform mixture. Then add in 48 grams of hexamine, and then rapidly stir the entire reaction mixture for about 1 hour. Thereafter, place a few pieces of steel wool (about 5 grams worth) into the reflux flask. Note: the steel wool should be torn into small pieces (about 1 centimeter cubes). Also, the steel wool should be totally clean, i.e, brand new and right from the bag. After adding the pieces of steel wool, reflux the entire reaction mixture at 100 Celsius for about 7 hours. During this reflux period, rapidly stir the reaction mixture. Note: a motorized stirrer works best, and is preferred over any other stirring means. After refluxing the reaction mixture for 7 hours, prepare a hot hydrochloric acid solution by placing 60 milliliters of 35 to 38% hydrochloric acid (31% muriatic acid will work) into a suitable sized beaker, and then add in 120 milliliters of water, and then quickly boil this acid mixture (a Bunsen burner works faster). When the acid mixture begins to boil around 100 Celsius, quickly pour it into the empty addition funnel on the reflux apparatus, and then rapidly add it (through the addition funnel) to the refluxing reaction mixture. After the addition of the acid solution, continue to reflux and rapidly stir the reaction mixture for an additional 10 minutes. Thereafter, remove the heat source and allow the reaction mixture to cool to room temperature. Then extract this entire reaction mixture with three 40-milliliter portions of diethyl ether, and after the extraction process, combine all ether portions (if not already done so), and then briefly wash this combined ether portion with one 50-milliliter portion of cold water. Note: after the extraction and single washing, the ether will be the upper layer each time. Now, place this washed combined ether portion into a clean beaker, and then slowly add in 66 grams of a 20% sodium hydroxide solution. The total sodium

hydroxide solution needed for the total extraction can be prepared by adding and dissolving 40 grams of sodium hydroxide into 150 milliliters of water. Note: sodium hydroxide generates excessive heat when dissolved in water, so allow the solution to cool before using. During the addition of the sodium hydroxide solution to the ether portion, rapidly stir the ether portion to ensure good mixing of the two-phase mixture. After the addition of the sodium hydroxide, continue to rapidly stir or blend the two-phase mixture for about 30 minutes. Thereafter, place the two-phase mixture into a seperatory funnel, and collect the upper ether layer (after removing the lower aqueous layer first). Note: if any insoluble solids exist at the bottom of the aqueous layer, filter the two-phase mixture before placing it into the seperatory funnel, and keep the filtered-off solids. Once the upper ether layer has been collected, repeat the extraction process with the 20% sodium hydroxide solution, two more times using 66 milliliters of the 20% sodium hydroxide solution per, and after each extraction, collect the upper ether layer, but filter-off any potential insoluble solids like before (if they exist), before using the seperatory funnel (just like before). After a total of three sodium hydroxide extractions have been carried out, you should now have some filtered-off solids, and an upper ether layer. Note: the lower aqueous layers can be recycled or discarded if desired. Now, to the upper ether layer, add to it, 100 milliliters of a 20% sodium hydroxide solution, and after the addition of the sodium hydroxide solution, rapidly blend the two-phase mixture for about 1 hour. Afterwards, filter-off any insoluble solids, and combine these filtered-off solids with any previously filtered-off solids. Then quickly wash all the combined filtered-off solids (which should be bright yellow crystals or powder) with two 25-milliliter portions of diethyl ether. After the washing process, vacuum dry or air-dry the solids. Now, dissolve the dry solids into 500 milliliters of water (note: more water may be needed—add more water if any yellowish solids still remain). After dissolving the yellowish solids into the water, quickly filter the solution to remove any non-yellowish, insoluble solids. Now, acidify this filtered aqueous solution by adding to it, 90 milliliters of a 20% hydrochloric acid solution. Then allow this entire acidified mixture to stand overnight at room temperature. The following day, filter-off the precipitated pale cream-colored crystals, wash them with several small portions of cold water, and then vacuum dry or air-dry the crystals. These crystals can then be recrystallized from 75 milliliters of petroleum ether to yield around 3.5 grams of pale yellow crystals of the desired product of eugenol aldehyde.

Step 2: Preparation of hydroxy eugenol

Into a suitable beaker or flask, add 3 grams of eugenol aldehyde (prepared in step 1), followed by 20 milliliters of pyridine. Then stir the entire mixture to form a uniform mixture. Thereafter, add in 23 milliliters of a 10% sodium hydroxide solution, and then slowly add in drop-wise, 12 milliliters of a 6% hydrogen peroxide solution, and during the addition, rapidly stir the reaction mixture. After the addition of the hydrogen peroxide, stir the reaction mixture for about 15 minutes, and then add in 50 milliliters of water to eliminate any turbidity. After adding in the water, continue to stir the reaction mixture for about 2 hours at room temperature. After 2 hours, add in 50 milliliters of a pre-chilled ice-cold 10% hydrochloric acid solution. After the addition of the acid, stir the entire reaction mixture for about 10 minute. Now, add in 50 grams of regular sodium chloride (table salt), and then rapidly stir the entire mixture to dissolve the bulk of the salt. Thereafter, extract the entire two-phase reaction mixture with three 30-milliliter portions of diethyl ether. After the extraction process, combine all ether portions (if not already done so), and then wash this combined ether portion with one 50 milliliter portion of 10% hydrochloric acid, followed by one 50 milliliter portion of a 5% sodium bicarbonate solution, followed finally by one 50 milliliter portion of ice cold 23% sodium chloride solution. Note: after the extraction and washing portions, the ether will be the upper layer each time. Then dry this washed ether portion by adding to it, 10 grams of anhydrous sodium sulfate, and then stir the entire oily mixture for 10 minutes—thereafter, filter-off the sodium sulfate. Finally, place the washed and filtered ether portion into a distillation apparatus, and distill-off the ether at 40 Celsius. When no more ether passes over or is collected, stop the distillation process, and recover the left over remaining oil (after it has cooled). Note: this oil can be purified by vacuum distillation at 176 Celsius under a vacuum of 20 milliliters of mercury if desired, but this process is not necessarily needed for step 3.

Step 3: Preparation of myristicin

Repeat steps 1 through 2, two or three more times to end up with at least 7.2 grams of the hydroxy eugenol compound. Then, into a suitable reflux apparatus (fitted with motorized stirrer or other stirring means, and thermometer), place 7.2 grams of the hydroxy eugenol compound (prepared in step 2), followed by 240 milliliters of dry acetone, followed by 9 milliliters of methylene bromide, and then followed by 36 grams of potassium carbonate. Thereafter, reflux the entire reaction mixture at 97 Celsius for about 22 hours. Note; during the reflux period, rapidly stir the reaction mixture. After refluxing the reaction mixture for 22 hours, quickly reduce the heat to 56 Celsius, and then quickly replace the reflux condenser with a standard cold-water condenser (fitted with a receiver flask). Then distill-off the acetone at 56 Celsius until no more acetone passes over or is collected. When this is achieved, remove the heat source, and allow the reaction mixture to cool to room temperature. Then recover the left over remaining residue, and place it into a suitable sized beaker. Then add in, 150 milliliters of cold water, and then rapidly blend the mixture for about 30 minutes. Thereafter extract this entire aqueous mixture with three 50-milliliter portions of diethyl ether, and after the extraction process, combine all ether portions (if not already done so). Note: after the extraction process, the ether will be the upper layer each time. Then place this combined ether portion into a suitable sized beaker, and then add in 100 milliliters of a 10% sodium carbonate solution, and then rapidly stir the entire mixture for about 10 minutes. Thereafter, recover the upper ether layer (by using a seperatory funnel), and then wash this upper ether layer with two

25-milliliter portions of ice-cold water. Note: after the washings with ice-cold water, the ether will be the upper layer each time. Then dry the collected ether portion by adding to it, 10 grams of anhydrous sodium sulfate. Then stir the entire mixture for about 10 minutes (to absorb water), and then filter-off the sodium sulfate. Finally, place this filtered ether portion into a distillation apparatus, and distill-off the ether at 40 Celsius. When no more ether passes over or is collected, stop the distillation process, and recover the left over remaining oil (after it has cooled). This oil can then be stored in amber glass bottles in a cool place until use. Note: this recovered oil can be purified by vacuum distillation at 138 Celsius under a vacuum of 10 milliliters of mercury, if desired.

Procedure B: Preparation of myristicin by isolation from nutmeg

Materials:

1. 50 grams of commercially available nutmeg butter	6. or 225 milliliters of methylene chloride
2. 500 milliliters of boiling 95% ethyl alcohol	7. or 10 grams of anhydrous magnesium sulfate
3. 100 milliliters of diethyl ether	8. or 200 milliliters of boiling 95% ethyl alcohol
4. 5 grams of anhydrous sodium sulfate	9. or 5 grams of anhydrous sodium sulfate
5. or 100 grams of powdered nutmeg (regular store bought nutmeg)	

Summary: Myristicin can be obtained in several ways, by isolation from various nutmeg products. In method 1, the myristicin can be obtained by extracting nutmeg butter with boiling alcohol, and then concentrating the resulting alcohol mixture by distillation. The concentrated alcohol mixture is then cooled, whereby crystals of the myristicin form and precipitate out. These crystals are then removed before they have chance to liquefy, and are then dissolved into ether. The ether is then briefly distilled to concentrate the mixture, and then chilled in the usual manner to induce crystallization of the myristicin. In method 2, myristicin can be obtained by first, steam distilling regular store bought nutmeg to produce the essential oil, oil of nutmeg, which is then recovered by using a seperatory funnel. The recovered oil of nutmeg is then extracted with solvent, and the solvent is then removed in the usual manner. Thereafter, a final purification is carried out by dissolving the left over oily residue (after evaporation of the extraction solvent) into boiling alcohol, filtering, and then chilling the alcohol mixture to induce crystallization of the dissolved myristicin. The crystallized myristicin is then easily filtered-off.

Hazards: Use care when handling diethyl ether, which is highly flammable, and capable of forming explosives mixtures with air—use proper ventilation and extinguish all flames before using.

Procedure:

Personnel notes for procedure B: Myristicin

Step 1: Isolation of myristicin from nutmeg butter (method 1)

Nutmeg butter is a product that is obtained by pressing nutmeg between heated plates in the presence of a small amount of steam. Nutmeg butter is composed primarily of myristicin, glycerides of myristic acid and other fats, and residue. The myristicin portion can be obtained by treating the nutmeg butter with ether or alcohol. To isolate myristicin from nutmeg butter, thoroughly mix 50 grams of commercially available nutmeg butter with 500 milliliters of boiling 95% ethyl alcohol. Note: make sure the 95% ethyl is boiling at 78 to 79 Celsius before adding in the nutmeg butter. While adding in the nutmeg butter, rapidly stir the boiling alcohol mixture, and after the addition of the nutmeg butter, place the entire alcohol mixture (including any and all insoluble solids) into a reflux apparatus (before the alcohol cools), and then reflux the entire mixture at about 79 Celsius for 2 hours. After 2 hours, quickly remove the reflux condenser, and replace it with a conventional condenser fitted with a receiver flask, and then distil-off the 95% ethyl alcohol until about 50% of the total volume remains (distill-off about 250 milliliters of the ethyl alcohol). When the alcohol mixture has been reduced to a total volume of about 50%, allow the alcohol concentrate to cool to about 60 Celsius, and then filter the entire alcohol mixture to remove any insoluble impurities. Note: this filtration process should be carried out before the alcohol mixture cools to below 60 Celsius. After the filtration process, place the entire filtered alcohol concentrate (even if two or more layers exist) into an ice bath, and chill it to about 0 Celsius. Note: a freezer can be used by itself or in combination with the ice bath. Then allow the alcohol concentrate to chill at 0 Celsius for about 2 hours. After 2 hours, filter-off the precipitated crystals of the myristicin (before the alcohol concentrate warms to above 5 Celsius), and then place these filtered-off crystals (before they have a chance to warm to above

10 celsius) into a suitable beaker, and then add in 100 milliliters of pre-heated diethyl ether (pre-heated to about 40 Celsius). Thereafter, stir the entire warm ether mixture for about 30 minutes, and then filter-off any insoluble impurities (if any). Then, place this warm ether mixture into a distillation apparatus, and distil-off the ether only until about 25% of the total volume has been reduced (distill-off only about 25 milliliters of ether). When 75% of the total ether volume remains, stop the distillation process, and then place the ether concentrate into an ice bath (before it cools), and then chill it to about 0 Celsius for about 1 hour. Note: a freezer can be used instead of an ice bath or in combination with. After chilling this ether concentrate to about 0 Celsius for 1 hour, filter the ether mixture to recover the crystallized myristicin (before it warms to above 5 Celsius), and then vacuum dry these filtered-off crystals of the myristicin (before they warm to above 5 celsius). Note: air dying will not work, and if desired, the myristicin can be dried by gently heating the crystals of the myristicin to induce liquefication, and then adding in 5 grams of anhydrous sodium sulfate (to absorb any moisture). After adding in the sodium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the sodium sulfate. The oil should then be stored in an amber glass bottle until use. Note: there are numerous modifications to this process, and those with experience should attempt any modifications they see fit.

Step 2: Isolation of myristicin from Oil of Nutmeg (method 2)

Into the steam distillation apparatus as illustrated in the following drawing, place 100 grams of powdered nutmeg (regular store bought nutmeg), followed by 750 milliliters of water. Thereafter, steam distill this mixture at 100 Celsius for about 4 to 6 hours. Note: the exact steam distillation process may vary, and should be continued until no more oily resinous material is seen collecting in the receiver flask. When no more oily resinous material is seen collecting in the receiver flask, stop the steam distillation process, and then recover the entire oily resinous aqueous mixture from the receiver flask, and then place this mixture into a beaker, and then gently heat to about 50 Celsius for about 10 minutes. Then, before the oily water mixture cools to below 50 Celsius, place it into a seperatory funnel, and then collect the upper oil layer. In some cases, the oil layer will be the bottom layer. Thereafter, extract this collected oil layer (before it cools to below 40 Celsius), with three 75-millilter portions of pre-heated methylene chloride (pre-heated to about 40 Celsius), and after the extraction process, combine all warm methylene chloride portions (if not already done so), and then dry this combined warm methylene chloride portion by adding to it, 10 grams of anhydrous magnesium sulfate. Note: after each extraction, the warm methylene chloride portion can be simply decanted-off rather then recovered by using a seperatory funnel. After adding in the anhydrous magnesium sulfate, stir the entire combined warm methylene chloride portion for about 10 minutes, and then filter-off the magnesium sulfate. Note: if during the stiring process (with the magnesium sulfate), the combined methylene chloride portion cools to below 30 Celsius, gently warm the entire mixture to 40 Celsius. Then place this warm methylene chloride portion into a distillation apparatus, and distill-off the methylene chloride at 40 Celsius. When no more methylene chloride passes over or is collected, stop the distillation process, and then recover the left over remaining oil (before it cools to below 40 Celsius). Immediately thereafter, dissolve this recovered warm oil into 200 milliliters of boiling 95% ethyl alcohol (pre-heated to about 78 celsius), and then quickly stir the entire alcohol mixture for about 5 minutes, and then filter-off any insoluble impurities (if any). Note: filter the alcohol mixture while its still boiling hot. After the filtration process, allow the alcohol mixture to slightly cool to about 60 Celsius, and then place it into an ice bath, and chill it to about 0 Celsius for about 2 hours. Note: a freezer can be used by itself or in combination with the ice bath. After chilling the alcohol mixture for about 2 hours, filter-off the crystallized myristicin, and then quickly vacuum dry this myristicin product (before it warms to above 5 Celsius). Note: air dying will not work, and if desired, the myristicin can be dried by gently heating the crystals of the myristicin to induce liquification, and then adding in 5 grams of anhydrous sodium sulfate (to absorb any moisture). After adding in the sodium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the sodium sulfate. The oil should then be stored in an amber glass bottle until use. Note: there are numerous modifications to this process, and those with experience should attempt any modifications they see fit.

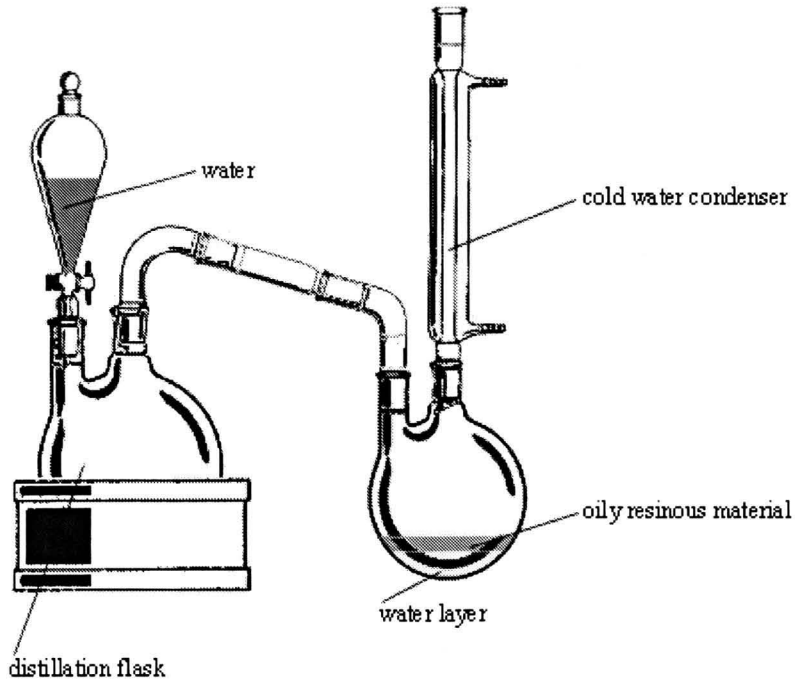
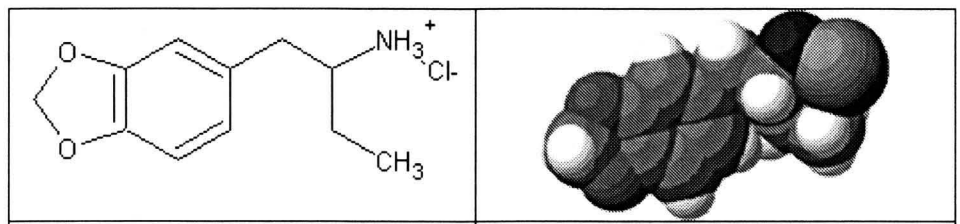


Figure 048. Steam distillation set-up for steam distilling nutmeg.

0027. BDB. 2-Amino-1-(3,4-methylenedioxyphenyl)butane hydrochloride. *1-(1,3-benzodioxol-5-yl)butan-2-amine hydrochloride*



Structure	3D model

BDB forms colorless to white crystals with a melting point of 161 Celsius. The impure crystals may have an off white to light brown tint with a melting point ranging from 158 to 160 Celsius. The crystals are soluble in water, but insoluble in ether and methylene chloride. BDB is very similar to MDMA in its properties and slightly in its physical effects on the body. The effects of BDB range from mild hallucinations, intoxification (however the intoxification is less then most other hallucinogenic/psychedelic amphetamines), feelings of well being, relaxation accompanied with periods of energy or fatigue, and a general sense of well being or sensation of good feeling. BDB does not produce the usual psychedelic effects that other similar compounds do, and it tends to produce a sense of "feeling good", similar to the effects of hydrocodone and codeine (of the pain killer category). The "good feelings" this drug produces is remarkable considering its psychedelic amphetamine like structure. Overall, BDB shows excellent potential for use as a mild stimulant, anti-depressant, mild hallucinogen, and "funny" drug—the latter as it relates to the overall good feelings it produces. Note: users of this drug should not drive cars or operate machinery while under the influence.

This substance is a controlled substance (hallucinogenic/amphetamine) as listed in the US code of Federal regulations.

Toxicity: Moderate (do not exceed a dose of 200 milligrams)	**Rate of onset (average):** Above moderate (symptoms begin within 30 minutes)
Stimulation dosage (ingestion): 150 to 175 milligrams	**Duration of effects (average):** 4 to 8 hours
Stimulation dosage (inhalation): 100 to 120	**Habit forming potential:** Moderate
Stimulation dosage (injection): unknown	**Estimated value U.S. (based on procedure):** $20 per gram.

Procedure A: Preparation of BDB

Materials:

1. 270 milliliters of dry diethyl ether	13. 113 milliliters of a 15% sulfuric acid solution
2. 4.6 grams of magnesium turnings	14. 600 milliliters of a 5% sodium hydroxide solution
3. 17.3 grams of dry 1-bromoprapane	15. 100 milliliters of a 23% sodium chloride solution
4. 16.6 grams of piperonal (see intermediate-0024. Piperonal)	16. 40 grams of anhydrous ammonium acetate
5. 25 milliliters of a 22% ammonium chloride solution	17. 3.1 grams of cyanoborohydride
6. 210 milliliters of a 5% hydrochloric acid solution	18. 20 drops of a 5% hydrochloric acid solution
7. 55 grams of anhydrous magnesium sulfate	19. 1000 milliliters of methylene chloride
8. 250 milligrams of finely divided potassium bisulfate	20. 400 milliliters of a 10% sulfuric acid solution
9. 60 milliliters of concentrated formic acid (80%+)	21. 40 grams of sodium hydroxide
10. 11.2 milliliters of a 35% hydrogen peroxide solution	22. 30 milliliters of 99% isopropyl alcohol
11. 60 milliliters of dry acetone	23. 15 grams of dry hydrogen chloride
12. 125 milliliters of methyl alcohol	

Summary: BDB is prepared in a three-step process starting with the formation of 1-(3,4-methylenedioxyphenyl)-2-butanol. This intermediate is prepared by reacting bromopropane with piperonal in a classic Grignard reaction whereby magnesium reacts with the bromopropane, forming the corresponding magnesium addition salt. This magnesium addition salt is then treated with the piperonal, which then reacts forming the desired intermediate. The desired intermediate is collected from the reaction mixture by treating the reaction mixture with ammonium chloride solution (to destroy magnesium salts), followed by dilute acid. The resulting reaction mixture is then separated into the solvent layer and water layer, and the solvent layer is then stripped. The resulting left over residue of 1-(3,4-methylenedioxyphenyl)-2-butanol is then converted into 1-(3,4-methylenedioxyphenyl)-2-butanone by reaction of 1-(3,4-methylenedioxyphenyl)-1-butene with formic acid and hydrogen peroxide. The reaction is stirred for several days, and then allowed to evaporate in the open. Note: the 1-(3,4-methylenedioxyphenyl)-1-butene is a pre-intermediate, and is prepared by dehydration of 1-(3,4-methylenedioxyphenyl)-2-butanol with potassium bisulfate. The desired 1-(3,4-methylenedioxyphenyl)-2-butanone is then collected from the evaporated reaction mixture, dissolved in methyl alcohol, treated with dilute acid, then recovered by extraction into solvent, and the solvent then evaporated. The left over remaining oily residue after removal of the solvent, is then converted into the desired BDB by reduction with cyanoborohydride in the presence of ammonium acetate and methyl alcohol. The reaction mixture is kept slightly acidic by the addition of dilute hydrochloric acid. After the reaction, the reaction mixture is treated with sodium hydroxide, and then extracted. The solvent extracts are then treated with acid, the resulting acid mixture is then made alkaline, and the resulting alkaline mixture is then extracted once again with solvent. The solvent extract is then finally evaporated, and the left over oily residue is then dissolved in isopropyl alcohol, treated with hydrogen chloride, and the desired BDB product is then precipitated by the addition of ether.

Hazards: As mentioned before, extinguish all flames before using diethyl ether, which is highly flammable and capable of forming explosive mixtures with air. Also, extinguish all flames before using acetone, methyl alcohol, and isopropyl alcohol. Methyl alcohol burns with a colorless flame. Store bromopropane in a amber glass bottle and away from sunlight, and avoid skin contact. Wear gloves when handling hydrochloric acid, sodium hydroxide, sulfuric acid, and concentrated formic acid—all of which are capable of causing skin irritation, especially concentrated formic acid. Handle concentrated hydrogen peroxide with care, and keep it away from combustible materials, alcohols, and ketones (such as acetone)—as explosive compounds are formed. Wear gloves when handling cyanoborohydride and avoid skin contact and contact with water and alcohol.

Procedure:

Personnel notes for procedure A: BDB

Step 1: Preparation of 1-(3,4-methylenedioxyphenyl)-2-butanol

Into a grignard reagent reflux apparatus (see laboratory tutorial for apparatus design), equipped with motorized stirrer, addition funnel, and two calcium chloride drying tubes (one attached on the top of the addition funnel, and the other attached to the top of the reflux condenser, place 20 milliliters of dry diethyl ether, followed by 4.6 grams of magnesium turnings. Thereafter, place 17.3 grams of dry 1-bromoprapane into the addition funnel. Note: the entire apparatus has to be absolutely dry before carryingout this reaction. Then, gradually add, drop-wise, the 1-bromopropane to the diethyl ether/magnesium turnings mixture over a period of about 15 to 30 minutes. Note: the time of addition depends on the temperature—as long as the temperature does not rise sporadically, addition of the 1-bromopropane should not take that long. During the addition, moderately stir the

diethyl ether/magnesium mixture, and maintain its temperature below 40 Celsius at all times. After the addition, continue to moderately stir the reaction mixture for about 10 minutes. Thereafter, quickly replace the addition funnel (just used), and replace it with a clean addition funnel (also fitted with calcium chloride drying tube), and then place into this addition funnel, a solution prepared by adding and dissolving 16.6 grams of piperonal into 70 milliliters of dry diethyl ether. Then add drop-wise, this piperonal solution over a period of about 30 minutes, while moderately stirring the reaction mixture. After the addition of the piperonal solution, reflux the entire reaction mixture at about 40 to 50 Celsius for about 3 hours. After 3 hours, remove the heat source, and allow the reaction mixture to cool to room temperature. Then pour the entire reaction mixture into a suitable sized beaker, and then place this beaker into an ice bath, and then add in 25 milliliters of a 22% ammonium chloride solution. During the addition, rapidly stir the reaction mixture. After the addition of the ammonium chloride solution, rapidly stir the entire mixture for about 30 minutes, and then filter-off the insoluble solids. Then place the filtered mixture into a seperatory funnel, and recover the upper ether layer. Then, wash this collected upper ether layer with three 70-milliliter portions of a 5% hydrochloric acid solution. Note: after each washing portion, the ether will be the upper layer each time. Then dry this collected washed ether layer by adding to it, 15 grams of anhydrous magnesium sulfate, and then stir the entire mixture for about 10 minutes—thereafter, filter-off the magnesium sulfate. Finally, place the filtered ether mixture into a distillation apparatus, and distill-off the ether at 40 Celsius. When no more ether passes over or is collected, stop the distillation process, and recover the left over remaining residue (after it has cooled), and then set it aside for step 2. Note: this left over residue can be purified by vacuum distillation at 98 Celsius under a vacuum of 0.070 millimeters of mercury, if desired.

Step 2: Preparation of 1-(3,4-methylenedioxyphenyl)-2-butanone

Into a suitable beaker or flask, place 16 grams of the product obtained in step 1, followed by 250 milligrams of finely divided potassium bisulfate. Then gently heat this mixture to 170 Celsius (using a small Bunsen burner flame, which works best), until no more water vapor is given off. Note: to determine when no more water vapor is produced, briefly stick a watch glass or other glass plate over the heated mixture—if any moisture condenses on the surface of the glass, continue heating until otherwise. Thereafter, remove the heat source, and allow the mixture to cool to room temperature. The result will be the pre-intermediate of 1-(3,4-methylenedioxyphenyl)-1-butene.

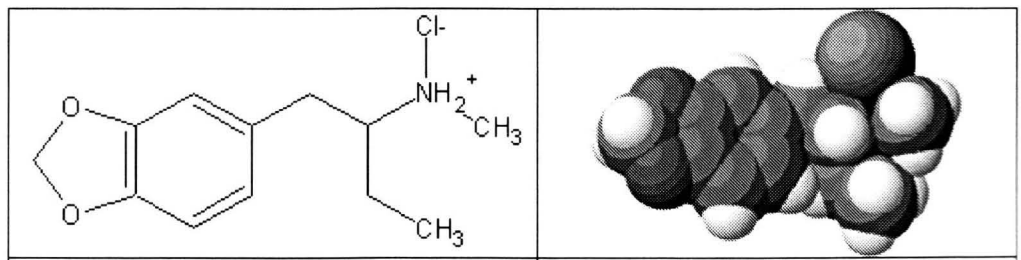
Now, into a suitable flask or beaker (equipped with motorized stirrer or other stirring means), place 60 milliliters of concentrated formic acid, and then chill this formic acid in an ice bath. When its internal temperature reaches about 5 Celsius, add in 8 milliliters of water, followed by slowly adding in 11.2 milliliters of a 35% hydrogen peroxide solution. Note: during the addition of the hydrogen peroxide, moderately stir the reaction mixture. Then quickly prepare a solution by adding and dissolving 13 grams of the product obtained in the first paragraph of step 2 into 60 milliliters of dry acetone, and then stir the entire mixture for about 10 minutes—thereafter, filter the acetone mixture to remove any insoluble impurities. Then add this filtered acetone solution to the formic acid/hydrogen peroxide mixture over a period of time sufficient to keep the reaction mixture below 40 Celsius at all times. Note: During the addition, rapidly stir the reaction mixture. After the addition, continue to stir the reaction mixture for about 2 days at room temperature or at a temperature around room temperature. Now, pour this entire reaction mixture into a large pan (with a high surface area), and allow the reaction mixture to evaporate. Note: blowing air over the surface using a conventional cooling fan may speed up the evaporation process. During the evaporation process, do not allow the reaction mixture to get above 40 Celsius. When practically all the liquids and volatiles have evaporated, recover the left over remaining residue, and then dissolve it into 25 milliliters of methyl alcohol. Thereafter, add in 113 milliliters of a 15% sulfuric acid solution, and then stir the entire mixture for about 10 minutes. Then place this mixture into a suitable sized reflux apparatus, and reflux the entire acidic mixture at 100 Celsius for about 1 hour. Afterwards, remove the heat source, and allow the refluxing acidic mixture to cool to room temperature. Thereafter, pour this cooled acidic mixture into a suitable sized beaker, and then extract this entire mixture with three 50-milliliter portions of diethyl ether, and after the extraction process, combine all ether portions (if not already done so), and then wash this combined ether portion with two 50-milliliter portions of cold water, followed by two 50-milliliter portions of a 5% sodium hydroxide solution, and then followed by two 50-millilter portions of a 23% sodium chloride solution. After the extraction and washings, the ether will be the upper layer each time. After the extraction and washings, dry the washed combined ether portion, by adding to it, 15 grams of anhydrous magnesium sulfate. Thereafter, stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Finally, place this filtered ether mixture into a distillation apparatus, and distill-off the diethyl ether at 40 Celsius. When no more ether passes over or is collected, stop the distillation process, and recover the left over remaining oily residue (after it has cooled), and keep for step 3. Note: purification of this oily residue can be accomplished by vacuum distillation at 135 Celsius under a vacuum of 0.30 millimeters of mercury, followed by re-distillation at 98 Celsius under a vacuum of 0.110 millimeters of mercury to obtain a colorless oil, but this vacuum distillation process is not generally needed for step 3.

Step 3: Preparation of BDB

Into a suitable flask or breaker, place 40 grams of anhydrous ammonium acetate, followed by 100 milliliters of methyl alcohol, and then followed by 9.2 grams of the product obtained in step 2. Then stir the entire mixture for about 10 minutes to form a uniform mixture. Thereafter, slowly add in 3.1 grams of cyanoborohydride over a period of time sufficient to keep the reaction

mixture from getting to hot (40 to 50 Celsius). Note: after addition of about 1/4th of the cyanoborohydride, add in about 5 drops of a 5% hydrochloric acid solution. After addition of ½ of the cyanoborohydride, add in another 5 drops of a 5% hydrochloric acid solution. Repeat this hydrochloric acid addition two more times—after 3/4ths, and after the final 1/4th portion of the cyanoborohydride. During the addition of the cyanoborohydride and resulting hydrochloric acid additions, rapidly stir the reaction mixture. After the addition of the cyanoborohydride and the following addition of the dilute hydrochloric acid, stir the entire reaction mixture for about 30 minutes, and then add in 500 milliliters of a 5% sodium hydroxide solution, and then stir the entire alkaline reaction mixture for 30 minutes. Thereafter, extract the entire reaction mixture with six 100-millliter portions of methylene chloride, and after the extraction process, combine all methylene chloride portions (if not already done so). Note: after the extraction process, the methylene chloride will be the lower layer each time. Then, extract the entire combined methylene chloride portion with four 100-milliliter portions of a 10% sulfuric acid solution, and after the extraction process, combine all aqueous sulfuric acid portions, if not already done so. Note: during the extraction, the aqueous sulfuric acid will be the lower layer each time—the upper methylene chloride layers can be recycled if desired. Thereafter, place the combined aqueous sulfuric acid portion into a suitable sized beaker, and then carefully and slowly add in a alkaline solution prepared by adding and dissolving 40 grams of sodium hydroxide into 100 milliliters of water. Note: sodium hydroxide generates excessive heat when dissolved in water, so allow the alkaline solution to cool before using. After the addition of the sodium hydroxide solution, rapidly stir the entire alkaline mixture for about 30 minutes. Then extract this entire alkaline mixture with four 100-millillter portions of methylene chloride, and after the extraction process, combine all methylene chloride portions (if not already done so), and then dry this combined methylene chloride portion by adding to it, 25 grams of anhydrous magnesium sulfate. Note: after the extraction process, the methylene chloride will be the lower layer each time. After adding in the magnesium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Finally, place the filtered methylene chloride portion into a distillation apparatus, and distill-off the methylene chloride at 40 Celsius. When no more methylene chloride passes over or is collected, stop the distillation process, and recover the left over remaining oily residue (after it has cooled). Then dissolve this oily residue into 30 milliliters of 99% isopropyl alcohol, and then filter the alcohol mixture to remove any insoluble materials. Thereafter, bubble into the alcohol mixture, 15 grams of dry hydrogen chloride gas (excess). After the addition of the hydrogen chloride, add in 30 milliliters of diethyl ether, and then stir the entire solvent mixture for about 10 minutes. Thereafter, filter-off the precipitated crystals of the desired BDB product, and then vacuum dry or air-dry the crystals.

0028. EDEN. 2-Methylamino-1-(3,4-methylenedioxyphenyl)butane hydrochloride. Methyl-J. *N-[1-(1,3-benzodioxol-5-ylmethyl)propyl]-N-methylamine; MBDB*



Structure	3D model

EDEN is a typical psychedelic amphetamine with good enhancements to sight, sound, taste, touch, and feelings. EDEN, like other similar compounds, forms colorless to whitish crystals, which may be colored amber to light brown when impure. The crystals have a melting point of 156 Celsius when pure, but the melting point may range from 150 to 157 when impure. EDEN demonstrates the usual psychedelic effects when administered orally in subjects—producing the usual enhancements to the surrounding world. Simple everyday activities such as getting the mail, watering the plants, and even taking a short drive are all enhanced with brilliant visuals, sensations, and a keen scope of mental awareness. Even though the drug produces a keen mental awareness, it still creates an intoxification that is similar to alcohol, but without the "alcohol" effects such as stupor, dizziness, drowsiness, wobbling or staggering movements, or other common alcohol intoxicating effects. The intoxification includes a superb array of feelings of well being, stimulation, other upper effects, and enhanced motion effects. EDEN is a fast acting drug, compared to most related compounds, and its effects can be felt within 15 to 20 minutes. The effects come on like a "rush", after the 15 to 20 minute mark. The rush effect is similar to an adrenaline rush, alcohol buzz, or feelings of goodness—the latter is similar to the high effect caused by a pain killer. Overall, EDEN demonstrates outstanding potential for use as a street drug, or therapeutic drug for the medical profession. The drug may cause dehydration, so water should be ingested on an hourly bases during the trip.

This substance is a controlled substance (psychedelic amphetamine) as listed in the US code of Federal regulations.

Toxicity: Low	**Rate of onset (average):** Above moderate (symptoms begin within 15 to 20 minutes)
Stimulation dosage (ingestion): 150 to 210 milligrams	**Duration of effects (average):** 4 to 6 hours

(best effects seem to come from 210 milligram dose)	
Stimulation dosage (inhalation): unknown	**Habit forming potential:** Low
Stimulation dosage (injection): unknown	**Estimated value U.S. (based on procedure):** $19 per gram.

Procedure A: Preparation of EDEN

Materials:

1. 340 milliliters of distilled water	9. 36.6 milliliters of a 25% sodium hydroxide solution
2. 10 grams of sodium hydroxide	10. 13.4 grams of 1-(3,4-methylenedioxyphenyl)-2-butanone (see 0027. BDB, step 2)
3. 20 grams of aluminum foil pieces	11. 50 milliliters of methyl alcohol
4. 100 milliliters of 95% ethyl alcohol	12. 200 milliliters of a 10% hydrochloric acid solution
5. 500 milligrams of mercury-II-chloride	13. 800 milliliters of methylene chloride
6. 260 milliliters of diethyl ether	14. 150 grams of a 25% sodium hydroxide solution
7. 30.4 grams of 50% methylamine hydrochloride solution	15. 20 grams of anhydrous magnesium sulfate
8. 184 milliliters of 99% isopropyl alcohol	16. 15 grams of dry hydrogen chloride gas

Summary: EDEN is prepared by the aluminum amalgamation of 1-(3,4-methylenedioxiyphenyl)-2-butanone. The reaction is carried out in isopropyl alcohol as the solvent, and in the presence of methylamine hydrochloride, and sodium hydroxide. The reaction is rather mild, and afterwards, the reaction is filtered, stripped of solvent, and the resulting left over residue is then dissolved into ether, treated with dilute acid, washed with solvent (to remove impurities), and then treated with base to liberate the purified freebase of the desired product. This purified freebase can then be dissolved into solvent (through extraction), and the resulting mixture then stripped of solvent. Finally, the left over residue is then dissolved into alcohol, and the desired product of EDEN then precipitated by the addition of hydrogen chloride gas.

Hazards: Extinguish all flames before using diethyl ether, which is highly volatile and flammable, and is capable of forming explosives mixtures with air. 95% ethyl alcohol, isopropyl alcohol, and methyl alcohol are flammable, and methyl alcohol burns with a colorless flame, so use caution. Wear gloves when handling mercury chloride, which is highly toxic and can be absorbed through the skin—use caution. Wear gloves when handling sodium hydroxide, and hydrochloric acid.

Procedure:

Personnel notes for procedure A: EDEN

Step 1: Amalgamation of aluminum

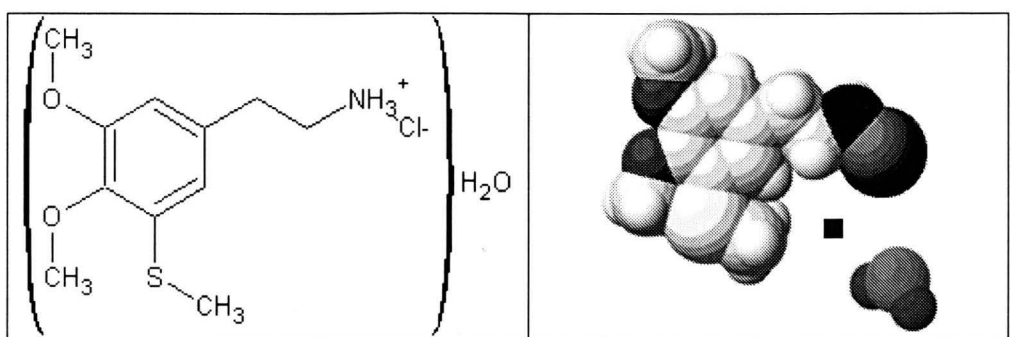
Into a suitable beaker, place 90 milliliters of distilled water, followed by 10 grams of sodium hydroxide. Thereafter stir the mixture to dissolve the sodium hydroxide. Note: much heat is generated when sodium hydroxide is dissolved in water, so allow the sodium hydroxide solution to cool to room temperature before using. Thereafter, add in 20 grams of aluminum foil pieces (cut into small squares), and allow the aluminum foil pieces to stand in the sodium hydroxide solution for about 20 minutes or until the evolution of hydrogen gas has drastically decreased. When the hydrogen gas evolution has almost ceased, filter-off the remaining pieces of aluminum, and then wash these collected pieces of aluminum with three 50-millilter portions of distilled water, followed by one portion of 50 milliliters of 95% ethyl alcohol (denatured alcohol can be used if desired). After the washing portion, allow the aluminum pieces to air-dry. When the pieces have air-dried, prepare a solution by adding and dissolving 500 milligrams of mercury-II-chloride (mercuric chloride) into 25 milliliters of water. Thereafter, add to the mercury chloride solution, the air-dried aluminum pieces, and allow the mixture to stand for about 15 minutes. After 15 minutes, filter-off the insoluble amalgated aluminum pieces, and then wash these filtered-off pieces with two 50-milliliter portions of distilled water, followed by one 50-milliliter portion of 95% ethyl alcohol (denatured alcohol will work if desired), and then wash with one portion of 10 milliliters of diethyl ether. After the washings, store the amalgated aluminum pieces submerged in a small amount of diethyl ether until use.

Step 2: Preparation of EDEN

First, take your aluminum amalgated pieces, stored submerged in a small amount of diethyl ether (prepared in step 1), and then quickly air-dry these pieces (10.2 grams total). Then, into a suitable sized beaker or flask (equipped with motorized stirrer or other stirring means), add the 10.2 grams of the air-dried amalgated aluminum pieces, followed by 30.4 grams of 50% methylamine hydrochloride solution, followed by 46 milliliters of 99% isopropyl alcohol, followed by 36.6 milliliters of a 25% sodium hydroxide solution, then followed by 13.4 grams of 1-(3,4-methylenedioxyphenyl)-2-butanone (see 0027. BDB, step 2), and then followed finally by 88 milliliters of 99% isopropyl alcohol. Then immediately place the beaker or flask into a cold water bath to keep the reaction mixture below 50 Celsius at all times. Then slowly stir the reaction mixture until the reaction is complete. When the reaction is complete, practically no shiny aluminum metal will remain, and only an insoluble gray sludge will remain. When most of the shinny metal has disappeared, and a gray sludge remains, filter the reaction mixture to remove the insoluble impurities, and then briefly wash the filtered-off solids with two 25-millilter portions of methyl alcohol. Note: after the washings, combine the methyl alcohol portions with the reaction mixture. Then, place this combined reaction mixture into a distillation apparatus, and remove all solvents by distillation at 100 Celsius (note: distillation under vacuum works best). When no more water, isopropyl alcohol, or methyl alcohol passes over or is collected, stop the distillation process, and recover the left over remaining (possible oily) residue (after it has cooled). Thereafter, dissolve the collected residue into 200 milliliters of diethyl ether, and then briefly filter the ether mixture to remove any insoluble impurities (if any). Then, extract this entire ether mixture with four 50-millilter portions of a 10% hydrochloric acid solution, and after the extraction process, combine all aqueous acid portions (if not already done so), and then discard or recycle the ether. Note: after the extraction process, the aqueous acidic portion will be the lower layer each time. Second note: if during the extraction process, any precipitates form, filter-off and save the precipitates as they will be crude crystals of the desired product. Then wash the combined aqueous acidic portion with three 100-millilter portions of methylene chloride. Note: after each washing, the methylene chloride will be the lower layer each time—afterwards, the methylene chloride can be recycled if desired. Second note: if any precipitates (crude crystals of the desired product) formed during the extraction of the ether where collected by filtration, briefly wash these filtered-off solids three times with the previously used methylene chloride portions used in washing the combined aqueous acidic portion. Thereafter, place any washed filtered-off solids (the washed crystals of the crude product) into a suitable beaker (if there where any), and then add in the washed combined aqueous acidic portion. Then add in, 150 grams of a 25% sodium hydroxide solution, and then rapidly blend the entire alkaline mixture for about 30 minutes. Afterwards, extract this entire alkaline mixture with ten 50-milliliter portions of methylene chloride, and after the extraction process, combine all methylene chloride portions (if not already done so), and then dry this combined methylene chloride portion by adding to it, 20 grams of anhydrous magnesium sulfate. Note: after each extraction, the methylene chloride will be the lower each time. After adding in the magnesium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Now, place the filtered methylene chloride portion into a distillation apparatus, and distill-off the methylene chloride at 100 Celsius. When no more methylene chloride passes over or is collected, stop the distillation process, and recover the left over remaining oily residue (after it has cooled). Finally, dissolve this oily residue into 50 milliliters of 99% isopropyl alcohol, and then stir the entire mixture for about 10 minutes. Then filter-off any insoluble solids, and then place this filtered isopropyl alcohol mixture into an ice bath, and chill to 0 Celsius. Then bubble into this alcohol mixture, 15 grams of dry hydrogen chloride gas. After the addition of the hydrogen chloride gas, stir the entire mixture for about 30 minutes, and then filter-off the precipitated crystals of EDEN, then wash the crystals with two 25-milliliter portions of diethyl ether, and then vacuum dry or air-dry the crystals. The result will be about 12 grams of the desired product of EDEN.

Note: other salts of the freebase EDEN can be prepared in the usual manner, by replacing the hydrogen chloride with 98% sulfuric acid, pure tartaric acid, citric acid, or 80% phosphoric acid.

0029. THIOMESCALINE. 3,4-dimethoxy-5-methylthiophenethylamine hydrochloride monohydrate. *5-[2-(chloroamino)ethyl]-1,2-dimethoxy-3-(methylthio)benzene hydrochloride monohydrate*



Structure | 3D model

THIOMESCALINE forms colorless to whitish crystals with a melting point 168 Celsius. The crystals are soluble in water and alcohol, but insoluble in ether, and methylene chloride. THIOMESCALINE is an interesting compound, which contains an active central sulfur atom rarely seen in most psychedelic amphetamines. THIOMESCALINE demonstrates some similarities to the original MESCALINE, but its psychological effects tend to linger on the side of pleasant feelings of bliss, rather then hallucinogenic sensations. The drug has been described as a "happy drug", inducing feelings and thoughts of happiness and pleasantry. Unlike the hallucinogenic and psychedelic nature of MESCALINE, THIOMESCALINE tends to be without the enhancements of sight, sounds, smell, touch, and feelings, and tends to produce the "happy" high with a corresponding intoxification that is very pleasant in nature. The happy high the drug produces is completely without any feelings of hate, anger, depression, nervousness, or unhappiness. The drug does however, produce a heightened sense of music, color, and mental awareness—some users have described an extra ordinary ability to write, or express other artistic ventures. In essence, THIOMESCALINE could be a considerable "happy" drug for treating severe cases of depression, anger, or other similar feelings, and as a mental awareness pill designed to heighten artistic imagination. Regardless of its happy or mental awareness characteristics, THIOMESCALINE produces an outstandingly good high that makes it well suitable for use as a street drug. Note: this compound may very well produce the best "high" of any of the psychedelic amphetamines. The drug has a low thresh-hold of addiction, it produces absolutely no headache, sickness or other withdrawal symptoms, and its side effects are very limited, and considered irrelevant.

This substance is a controlled substance (psychedelic amphetamine) as listed in the US code of Federal regulations.

Toxicity: Low	**Rate of onset (average):** Above moderate
Stimulation dosage (ingestion): 80 milligrams (best effects come from 80 milligram doses). Note: 100 milligram doses may produce paranoia and irritability	**Duration of effects (average):** 8 to 12 hours
Stimulation dosage (inhalation): unknown	**Habit forming potential:** Low
Stimulation dosage (injection): unknown	**Estimated value U.S. (based on procedure):** $29 per gram. Based on a $6 hit (80 milligram hit) = $75 per gram

Procedure A: Preparation of THIOMESCALINE

Materials:

1. 228 milliliters of glacial acetic acid	13. 240 milliliters of a 5% sodium hydroxide solution
2. 24 grams of sodium thiocyanate	14. 30 grams of anhydrous magnesium sulfate
3. 18 grams of vanillin (see intermediate-0019. 3,4,5-TMB, Procedure B, step 1)	15. 200 milliliters of nitromethane
4. 19 grams of liquid bromine	16. 5 grams of anhydrous ammonium acetate
5. 36 milliliters of a 5% hydrochloric acid solution	17. 10 grams of lithium aluminum hydride
6. 960 milliliters of 95% ethyl alcohol	18. 450 milliliters of tetrahydrofuran (THF)
7. 330 milliliters of methyl alcohol	19. 8 milliliters of 98% sulfuric acid
8. 70 grams of methyl iodide	20. 150 milliliters of a 15% sodium hydroxide solution
9. 67 grams of sodium hydroxide	21. 1150 milliliters of diethyl ether
10. 120 milliliters of dimethylsulfoxide (DMSO)	22. 480 milliliters of a 10% sulfuric acid solution
11. 50 grams of 35 to 38% hydrochloric acid	23. 80 milliliters of cold 99% isopropyl alcohol
12. 870 milliliters of methylene chloride	24. 15 grams of dry hydrogen chloride gas

Summary: THIOMESCALINE is prepared in a three step process starting with the formation of 5-formyl-7-methoxy-2-oxo-1,3-benzoxathiole. This intermediate is prepared by reacting the readily available vanillin with sodium thiocyanate in the presence of glacial acetic acid and bromine. The reaction is rather general and afterwards, it is quenched with dilute acid, followed by ethyl alcohol. The resulting mixture is then briefly heated, and then diluted with water to induce precipitation of the desired crude product. The crude product is then filtered-off, and then recrystallized from fresh alcohol. The purified crystals of the desired 5-formyl-7-methoxy-2-oxo-1,3-benzoxathiole is then converted into 3,4-dimethoxy-5-(methylthio)benzaldehyde by refluxing with methyl iodide in methyl alcohol and in the presence of sodium hydroxide. After the reaction, the mixture is evaporated, and the left over residue is then treated with a DMSO solution of methyl iodide. Shortly thereafter, additional methyl iodide is added, along with a small amount of sodium hydroxide, and the resulting reaction mixture is then drowned into cold water, acidified with dilute hydrochloric acid, and then extracted with methylene chloride. The methylene chloride extract is then washed, dried, and then evaporated to leave behind the crude desired product, which can be purified by recrystallization to afford a refined product of 3,4-dimethoxy-5-(methylthio)benzaldehyde. This 3,4-dimethoxy-5-(methylthio)benzaldehyde is then converted into the pre-intermediate of 3,4-dimethoxy-5-methoxy-beta-nitrostyrene by reaction with excess nitromethane in the presence of a small amount of anhydrous ammonium acetate as catalyst. The reaction is very general, and after refluxing it for several hours, the excess solvent is driven off, and then the resulting dry residue is then dissolved into hot solvent, and upon cooling of this solvent, the desired pre-intermediate precipitates as yellowish crystals, which are then easily filtered-off. These crystals of the nitro styrene are then finally converted into the desired product of

THIOMESCALINE by reaction with lithium aluminum hydride in tetrahydrofuran in the presence of sulfuric acid. Thereafter, the reaction mixture is refluxed briefly, and then diluted with water to destroy any excess lithium salt. The reaction mixture is then acidified, basified, and extracted in the usual manner. The solvent extracts are then evaporated, and the resulting left over residue is then dissolved into alcohol, and the desired product is then precipitated in the usual manner by the addition of hydrogen chloride.

Hazards: Wear gloves when handling glacial acetic acid, hydrochloric acid, sulfuric acid, and sodium hydroxide—all are capable of causing skin irritation. Extinguish all flames before using diethyl ether, tetrahydrofuran, and nitromethane, all of which are highly flammable and can form explosive mixtures with air. Wear gloves and use maximum ventilation when handling liquid bromine, which is highly toxic and very irritating to the eyes, nose, and throat. Wear gloves and use caution when handling lithium aluminum hydride, and avoid contact with water. Store methyl iodide in amber glass bottles away from sunlight to prevent deterioration. Use caution when handling methyl alcohol, and avoid contact with flames, sparks, electric discharges and other methods of ignition—as methyl alcohol burns with a colorless flame.

Procedure:

Personnel notes for procedure A: Thiomescaline

Step 1: Preparation of 5-formyl-7-methoxy-2-oxo-1,3-benzoxathiole

Into a suitable flask or beaker (equipped with motorized stirrer or other stirring means, and thermometer), place 180 milliliters of glacial acetic acid, followed by 24 grams of sodium thiocyanate, and then followed by 18 grams of vanillin. Thereafter, stir the entire mixture for about 10 minutes to form a uniform mix. Then place this mixture into an ice bath, and chill to 0 Celsius. While the mixture is chilling to 0 Celsius, prepare a bromine solution by adding and dissolving 19 grams of liquid bromine into 48 milliliters of glacial acetic acid. When the temperature of the vanillin mixture reaches about 0 Celsius, slowly add drop-wise, the bromine solution over a period of about 18 minutes while rapidly stirring the vanillin mixture and maintaining its temperature below 5 celsius. After the addition of the bromine solution, immediately add in 36 milliliters of a 5% hydrochloric acid solution, followed by 360 milliliters of 95% ethyl alcohol. After both additions, continue to rapidly stir the reaction mixture for an additional 36 minutes. After stirring for about 36 minutes, briefly but gently heat the entire reaction mixture to 78 Celsius with stirring. When the reaction mixture reaches 78 Celsius, quickly stop the heating process, and immediately filter the reaction mixture while it's still hot. After filtering-off any insoluble materials, allow the filtered reaction mixture to cool to room temperature. Thereafter, quickly filter the reaction mixture to remove any insoluble impurities that may have precipitated during the cooling process (if any). Then dilute the filtered reaction mixture by adding to it, 550 milliliters of cold water, and then stir the entire diluted reaction mixture for about 30 minutes. Then filter-off the precipitated crystals of the desired product, and then vacuum dry or air-dry the filtered-off solids. Then recrystallize these filtered-off solids from 300 milliliters of fresh 95% ethyl alcohol, and after the recrystallization process, vacuum dry or air-dry the filtered-off solids.

Step 2: Preparation of 3,4-dimethoxy-5-(methylthio)benzaldehyde

Into a suitable reflux apparatus (equipped with motorized stirrer or other stirring means, and thermometer), place 15 grams of the product obtained in step 1, followed by 120 milliliters of methyl alcohol, and then followed by 34 grams of methyl iodide. Then blend the entire mixture for about 10 minutes to form a uniform mix. Thereafter, add in a sodium hydroxide solution prepared by adding and dissolving 14 grams of sodium hydroxide into 120 milliliters of methyl alcohol. Note; sodium hydroxide generates heat when dissolved in methyl alcohol, but never mind this and add the warm alkaline methyl alcohol mixture after preparation. After adding in the alkaline alcohol mixture, reflux the entire reaction mixture at 68 Celsius for about 70 minutes. After refluxing the reaction mixture for about 70 minutes, quickly replace the reflux condenser with a conventional cold-water condenser (fitted with receiver flask), and then distill-off the methyl alcohol at 68 Celsius. When no more methyl alcohol passes over or is collected, stop the distillation process, and then recover the left over residue (after it has cooled). Then place this left over residue into a suitable sized beaker, and then add in a solution prepared by adding and dissolving 17 grams of methyl iodide into 120 milliliters of dimethylsulfoxide (DMSO), and then stir the entire mixture for about 70 minutes. After 70 minutes, add in 3 grams of sodium hydroxide, followed by 19 grams of methyl iodide. After both additions, continue to stir the entire mixture for about 144 minutes. Afterwards, pour the entire mixture into 960 milliliters of cold water, and thereafter, add in 50 grams of 35 to 38% hydrochloric acid. After the addition of the acid rapidly stir the entire acidic mixture for about 30 minutes. Thereafter, extract the entire acidic mixture with three 90-milliliter portions of methylene chloride, and after the extraction process, combine all methylene chloride portions (if not already done so), and then wash this combined methylene

chloride portion with three 80-milliliter portions of a 5% sodium hydroxide solution, followed by three 50-milliliter portions of cold water. Note: after the extraction and washing portions, the methylene chloride will be the lower layer each time. After the washings, dry the methylene chloride portion by adding to it, 15 grams of anhydrous magnesium sulfate, and then stir the entire mixture for about 10 minutes—thereafter, filter-off the magnesium sulfate. Finally, place the filtered methylene chloride portion into a distillation apparatus, and distill-off the methylene chloride at 40 Celsius. When no more methylene chloride passes over or is collected, stop the distillation process, and recover the left over remaining residue (after it has cooled). Then recrystallize this left over remaining residue from 300 milliliters of 95% ethyl alcohol, and after the recrystallization process, vacuum dry or air-dry the filtered-off solids.

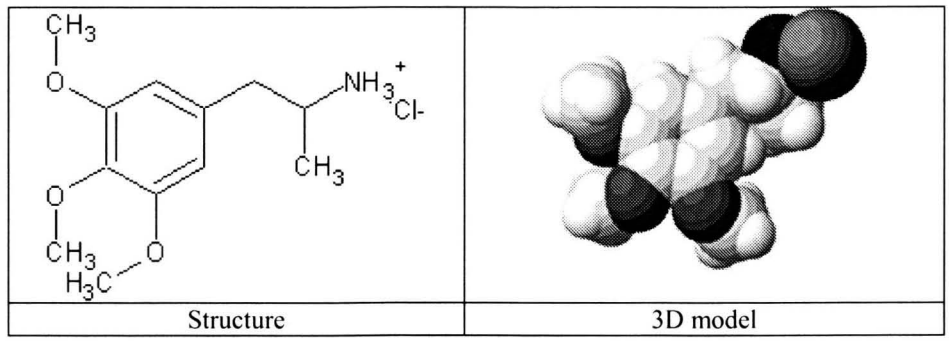
Step 3: Preparation of THIOMESCALINE

Into a standard reflux apparatus (equipped with motorized stirrer or other stirring means, and thermometer), place 9 grams of the product obtained in step 2, followed by 200 milliliters of nitromethane, and then followed by 5 grams of anhydrous ammonium acetate. Thereafter, reflux the entire mixture at 102 Celsius for about 8 hours. During the reflux process, rapidly stir the mixture. After refluxing the entire mixture for about 8 hours, quickly remove the reflux condenser, and replace it with a conventional condenser fitted with a receiver flask, and then distill-off the large excess of nitromethane at 102 Celsius. When no more nitromethane passes over or is collected, stop the distillation process, and recover the left over reaming residue (after it has cooled). Thereafter, dissolve this brownish left over residue into 40 milliliters of hot methyl alcohol. Thereafter, allow the methyl alcohol solution to cool to room temperature. Then filter-off the precipitated yellowish crystals, wash them with two 25-milliliter portions of ice-cold methyl alcohol, and then vacuum dry or air-dry the filtered-off crystals. The result will be about 4 grams of the pre-intermediate nitro styrene with a melting point of 120 Celsius.

Into a reflux apparatus fitted with motorized stirrer, thermometer, and addition funnel, place 10 grams of lithium aluminum hydride, followed immediately by 250 milliliters of tetrahydrofuran (THF). Note: the top of the reflux condenser needs to be fitted with a calcium chloride drying tube. Afterwards, place the flask containing the lithium aluminum hydride/THF mixture into an ice bath, and chill to 0 Celsius. Then place 8 milliliters of 98% sulfuric acid into the addition funnel, and then immediately thereafter, add it drop-wise, to the lithium aluminum hydride/THF mixture over a period sufficient to keep the lithium aluminum hydride/THF mixture below 5 Celsius at all times. Immediately thereafter, add to the same addition funnel, a solution prepared by adding and dissolving 7 grams of the pre-intermediate nitro styrene (obtained in the first paragraph of this third step), into 100 milliliters of tetrahydrofuran. Then add drop-wise, this mixture to the lithium aluminum hydride/THF mixture over a period sufficient to keep the reaction mixture below 5 Celsius. After the addition, reflux the entire reaction mixture at 68 Celsius for about 10 minutes, and after refluxing for about 10 minutes, remove the heat source and allow the reaction mixture to cool to room temperature. Then place the entire reaction mixture into a suitable sized beaker, and then add in a solvent mixture prepared by adding and dissolving 150 milliliters of water into 50 milliliters of tetrahydrofuran. Then rapidly stir the entire reaction mixture for about 5 minutes, and then slowly add in, drop-wise, 150 milliliters of a 15% sodium hydroxide solution. Thereafter, stir the entire reaction mixture for about 10 minutes. Then filter the alkaline reaction mixture to remove any insoluble impurities, and then quickly wash these filtered-off solids with two 25-milliliter portions of fresh tetrahydrofuran. Then combine these two tetrahydrofuran washing portions with the filtered reaction mixture, and then dilute this total combined reaction mixture by adding to it, 650 milliliters of diethyl ether. Then extract this entire mixture with twelve 40-milliliter portions of a 10% sulfuric acid solution, and after the extraction process, combine all dilute sulfuric acid portions (if not already done so), and then wash this combined dilute sulfuric acid portion with two 100-milliliter portions of diethyl ether. Note: after the extraction and washing portions the dilute sulfuric acid portion will be the lower layer each time. Thereafter, place this washed combined dilute sulfuric acid portion into a suitable sized beaker, and then slowly add in a sodium hydroxide solution prepared by adding and dissolving 50 grams of sodium hydroxide into 200 milliliters of water. Note: sodium hydroxide generates excessive heat when dissolved in water, so allow the solution to cool to room temperature before using. After the addition of the sodium hydroxide solution, rapidly stir the entire alkaline mixture for about 30 minutes. Thereafter, extract this entire alkaline mixture with twelve 50-milliliter portions of methylene chloride, and after the extraction process, combine all methylene chloride portions (if not already done so), and then dry this combined methylene chloride portion by adding to it, 15 grams of anhydrous magnesium sulfate. Note: after the extraction process, the methylene chloride will be the lower layer each time. After adding the anhydrous magnesium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Finally, place the filtered methylene chloride portion into a distillation apparatus, and distill-off the methylene chloride at 40 Celsius. When no more methylene chloride passes over or is collected, stop the distillation process, and recover the left over remaining residue (after it has cooled). Then, dissolve this residue into 80 milliliters of cold 99% isopropyl alcohol, and then stir the entire mixture for about 10 minutes, and then filter the alcohol mixture to remove any potential insoluble impurities. Thereafter, bubble into this alcohol mixture, 15 grams of dry hydrogen chloride gas (excess), and after the addition of the hydrogen chloride, add in 300 milliliters of diethyl ether. Then let the entire mixture stand for about 30 minutes, and then filter-off the precipitated crystals of the desired product, and then vacuum dry or air-dry the filtered-off crystals of THIOMESCALINE.

Note: other salts of the freebase THIOMESCALINE can be prepared in the usual manner, by replacing the hydrogen chloride with 98% sulfuric acid, pure tartaric acid, citric acid, or 80% phosphoric acid.

0030. TMA. 3,4,5-Trimethoxyamphetamine hydrochloride. *1-(3,4,5-trimethoxyphenyl)propan-2-amine hydrochloride*



Structure	3D model

TMA forms colorless to whitish crystals that may have a pink tint when impure—the crystals have a melting point of 211 Celsius, but impure TMA has a melting point ranging from 194 to 212 Celsius. The crystals are soluble in water and alcohol but insoluble in ether and methylene chloride. In short, TMA is very similar to MESCALINE, but without the nausea or vomiting often associated with the consumption of MESCALINE. TMA produces similar effects and psychedelic attributes to MESCALINE, but with a slight twist ranging in the areas of psychiatrics. In essence, the drug tends to give users the ability to analyze their own psychic mentality, making the drug very useful for use in psychiatric studies for the medical community. The MESCALINE like similarities of TMA include the usual psychedelic enhancement to sight, sound, touch, smells, feelings, ect., ect., Secondary effects include increased mental awareness, strange color enchantments, visual aids and visual enhancements, a heightened sense of music and other sounds, and motion. In some cases TMA may produce nausea or vomiting in the first hour of ingestion, and may cause irritability. Note: TMA was the first synthetic psychedelic amphetamine synthesized—synthesized in 1955 by Canadian chemists.

This substance is a controlled substance (psychedelic amphetamine) as listed in the US code of Federal regulations.

Toxicity: Low	**Rate of onset (average):** Moderate
Stimulation dosage (ingestion): 135 milligrams	**Duration of effects (average):** 6 to 8 hours
Stimulation dosage (inhalation): unknown	**Habit forming potential:** Low
Stimulation dosage (injection): unknown	**Estimated value U.S. (based on procedure):** $25 per gram

Procedure A: Preparation of TMA

Materials:

1. 13 grams of 3,4,5-trimethoxybenzaldehyde (see intermediate –0019. 3,4,5-TMB	9. 9.5 grams of lithium aluminum hydride
2. 110 milliliters of pre-heated warm 95% ethyl alcohol (pre-heated to about 40 Celsius)	10. 185 milliliters of anhydrous diethyl ether
3. 5.2 grams of nitroethane	11. 390 milliliters of dry tetrahydrofuran
4. 500 milligrams of n-butyl amine	12. 25 milliliters of a 15% sodium hydroxide solution
5. or 10 grams of 3,4,5-triemthoxybenzaldehyde (see intermediate-0019. 3,4,5-TMB)	13. 15 grams of dry hydrogen chloride gas
6. or 37 milliliters of nitroethane	14. 40 milliliters of 99% isopropyl alcohol
7. or 2 grams of anhydrous ammonium acetate	15. 100 milliliters of acetonitrile
8. or 200 milliliters of boiling methyl alcohol	

Summary: TMA is produced in a rather simple two-step process starting with the formation of the nitro intermediate of 3,4,5-triemthoxybenzaladehye. This nitro intermediate is readily prepared by condensing 3,4,5-trimethoxybenzaldehdye with nitroethane in the presence of 95% ethyl alcohol and a small amount of the catalyst n-butyl amine. The reaction is allowed to stand for several days, where upon the mixture is chilled, and the dissolved nitro intermediate precipitates as yellowish crystals. Recrystallization of these yellowish crystals affords the desired nitro intermediate. In another method of preparation, the nitro intermediate can be prepared by condensing 3,4,5-trimethoxybenzaldehdye with nitroethane in the presence of anhydrous ammonium acetate as catalyst. The reaction is similar to method 1, but refluxing is used to carryout the reaction. After the reaction is complete, the mixture is evaporated, and the left over oily residue is then dissolved into boiling methyl alcohol, and upon cooling, crystals of the nitro intermediate separate out. These crystals can then be purified by recrystallization from fresh

methyl alcohol. In either case, the nitro intermediate can then be converted into the desired TMA by reaction with the usual reducing agent of lithium aluminum hydride. The reaction is carried out in the usual manner, and after the reduction with the lithium salt is complete, the reaction mixture is treated with water and base to destroy any lithium salt, filtered, and then evaporated. The left over oily residue is then dissolved into alcohol, and the desired TMA is then formed by the addition of hydrogen chloride. The crude TMA is then precipitated by the addition of diethyl ether, and the crude crystals are then purified by soaking them with acetonitrile.

Hazards: Wear gloves when handling acetonitrile, which is toxic and can be absorbed through the skin. Extinguish all flames before using diethyl ether, tetrahydrofuran, and nitroethane, all of which are highly flammable and can form explosive mixtures with air. Wear gloves and use caution when handling lithium aluminum hydride, and avoid contact with water. Use caution when handling methyl alcohol, and avoid contact with flames, sparks, electric discharges and other methods of ignition—as methyl alcohol burns with a colorless flame.

Procedure:

Personnel notes for procedure A: TMA

Step 1: Preparation of the nitro intermediate of 3,4,5-trimethoxybenzaldehyde (method 1)

Into a suitable flask or beaker, place 13 grams of 3,4,5-trimethoxybenzaldehyde, followed by 10 milliliters of pre-heated 95% ethyl alcohol (pre-heated to about 40 Celsius). Thereafter, briefly stir the entire mixture for about 10 minutes to form a uniform mixture. Then add in 5.2 grams of nitroethane, followed by 500 milligrams of n-butyl amine. Then allow the entire reaction mixture to stand at 40 Celsius for 3 days. Note: a hot plate or other external heating source may be needed to keep the reaction mixture at 40 Celsius for 3 days. After 3 days, place the reaction mixture into an ice bath, or freezer, and chill to about 0 Celsius for several hours. Note: during the chilling process, occasionally scratch the side of the flask or beaker with a glass stir rod to invoke crystallization of the dissolved nitro intermediate. After allowing the reaction mixture to stand at 0 Celsius for several hours, filter-off the precipitated crystals of the nitro intermediate, and then vacuum dry or air-dry them. Then recrystallize these dried crystals from 100 milliliters of 95% ethyl alcohol, and after the recrystallization process, vacuum dry or air-dry the filtered-off yellowish crystals. The result will be about 15 grams of 2-nitro-1-(3,4,5-trimethoxyphenyl)propene as yellowish crystals with a melting point of 95 Celsius.

Step 1: Preparation of the nitro intermediate of 3,4,5-trimethoxybenzaldehyde (method 2)

Into a suitable reflux apparatus, place 10 grams of 3,4,5-triemthoxy benzaldehyde, followed by 37 milliliters of nitroethane. Thereafter, briefly stir the entire mixture to form a uniform mixture. Thereafter, add in 2 grams of anhydrous ammonium acetate, and then reflux the entire mixture at 115 Celsius until the reaction mixture turns red. When the reaction mixtures turn red, stop the reflux process, and allow the reaction mixture to cool to room temperature. Then pour the entire reaction mixture into a large shallow pan, and allow the reaction mixture to air evaporate. Note: evaporating the reaction mixture under vacuum and under a temperature of 80 Celsius works best, and affords the recovery of the un-reacted nitroethane. If using a shallow pan, blowing air over the surface of the reaction mixture using a conventional cooling fan helps speed up the evaporation process. When the reaction mixture has been evaporated, and only an oily residue remains, recover this left over oily residue, and then dissolve it into 100 milliliters of boiling methyl alcohol. Thereafter, remove the heat source, ands allow the methyl alcohol to cool to room temperature. On cooling to room temperature, some yellowish crystals of the nitro intermediate will crystallize out. Then place this methyl alcohol mixture into an ice bath, and chill to about 0 Celsius. Then allow this methyl alcohol mixture to stand at 0 Celsius for about 1 hour, and then filter-off the precipitated yellowish crystals of the nitro intermediate—then vacuum dry or air-dry these filtered-off crystals. Finally, recrystallize these dried crystals from 100 milliliters of methyl alcohol, and after the recrystallization process, vacuum dry or air-dry the filtered-off collected crystals.

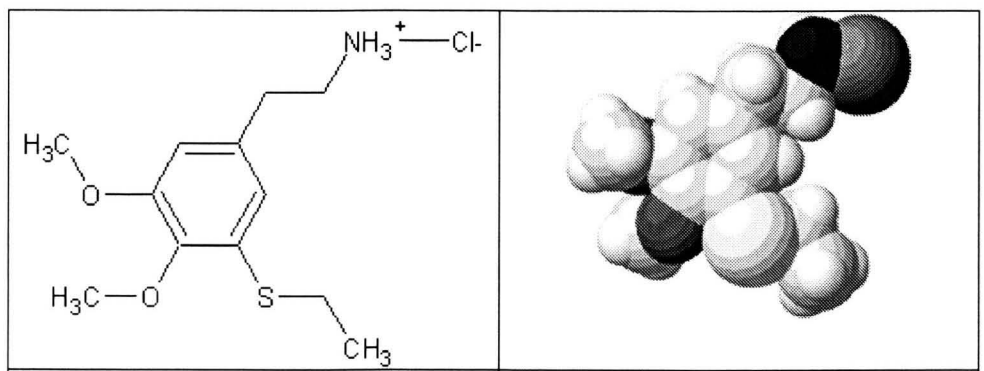
Step 2: Preparation of TMA

Into a standard reflux apparatus (fitted with addition funnel, thermometer, and motorized stirrer or other stirring means), place 9.5 grams of lithium aluminum hydride, followed immediately by 25 milliliters of anhydrous diethyl ether, and then followed by 250 milliliters of dry tetrahydrofuran. Then gently warm this mixture to about 40 Celsius. Then, into the addition funnel, place a solution prepared by adding and dissolving 11 grams of the product obtained in step 1, into 40 milliliters of tetrahydrofuran. Then add this solution drop wise, to the lithium aluminum hydride mixture over a period sufficient to keep the

reaction mixture below 50 Celsius at all times. After the addition, stir the reaction mixture and maintain a gentle reflux at 40 to 50 Celsius for about 9 hours. After 9 hours, stop the reflux process, and allow the reaction mixture to cool to room temperature. Then place the cooled reaction mixture into an ice bath, and then slowly and carefully add in, 10 milliliters of cold water, followed by a slow and careful addition of 25 milliliters of a 15% sodium hydroxide solution, and then finally by the careful addition of 30 milliliters of cold water. Thereafter, stir the entire diluted reaction mixture for about 10 minutes, and then filter-off the insoluble materials formed. Then quickly wash these filtered-off solids with two 50-millilter portions of tetrahydrofuran, and after the washing process, combine these two tetrahydrofuran portions with the filtered reaction mixture. Then place this filtered combined reaction mixture into a distillation apparatus, and distill-off all the liquids at 100 Celsius. When no more diethyl ether, tetrahydrofuran, and water passes over or is collected, stop the distillation process, and then recover the left over remaining oily residue (after it has cooled). Then dissolve this left over oily residue into 40 milliliters of 99% isopropyl alcohol, and then briefly stir this alcohol mixture for about 30 minutes. Then filter this alcohol mixture to remove any insoluble materials, and then bubble into this filtered alcohol mixture, 15 grams of dry hydrogen chloride gas (excess). After the addition of the hydrogen chloride gas, stir the entire acidic alcoholic mixture for about 10 minutes, and then add in 160 milliliters of diethyl ether. After the addition of the diethyl ether, moderately stir the entire mixture for about 1 hour at room temperature. After 1 hour, filter-off the precipitated crystals, and then vacuum dry or air-dry these crystals. Finally, thoroughly blend these dried crystals with 75 milliliters of acetonitrile for about 1 hour, and then filter the acetonitrile mixture to recover the white crystals of the desired product of TMA. These crystals can then be washed with two 25-millilter portions of fresh cold acetonitrile, and then vacuum dried or air-dried.

Note: other salts of the freebase TMA can be prepared in the usual manner, by replacing the hydrogen chloride with 98% sulfuric acid, pure tartaric acid, citric acid, or 80% phosphoric acid.

0031. THIOESCALINE. Thioethylmescaline. 3-Thiometaescaline. 4,5-Dimethoxy-3-ethylthiophenethylamine hydrochloride. *2-[3-(ethylthio)-4,5-dimethoxyphenyl]ethanamine hydrochloride*



Structure | 3D model

THIOESCALINE forms white to off-white crystals with a melting point of 172 Celsius. The crystals are soluble in water and alcohol, but insoluble in ether and methylene chloride. THIOESCALINE is a strange psychedelic amphetamine with unusual psychological activity. The psychological activity is un-like the other psychedelic amphetamines, and tends to be more "passive" in nature. This passive natured high focuses heavily on the spiritual and emotional side of the mind rather then on physical enhancements of sight, sound, touch ect., ect., experienced with the more common psychedelic amphetamines. In essence, many have described this compound as being a "religious" or spiritual drug. The high experienced by users has been summed up as being a "pleasant trip", with heavy enhancements to spiritual, and emotional thought. The passive natured psychological activity of this drug has warranted its research by the government for use in chemical warfare and in prisoner interrogations—also known as "truth" serums. Although THIOESCALINE is much different then most other psychedelic amphetamines, the high it produces is good enough and adequate for mass consumption by customers, and its use has been heavily encouraged by many religious leaders as a method of "discovering" God.

This substance is a controlled substance (psychedelic amphetamine) as listed in the US code of Federal regulations.

Toxicity: Low	**Rate of onset (average):** Below moderate (may take up to 90 minutes for effects to be realized)
Stimulation dosage (ingestion): 60 to 100 milligrams	**Duration of effects (average):** 10 to 15 hours (Note: some users have stated the effects lasted up to 3 days)
Stimulation dosage (inhalation): 40 to 50 milligrams	**Habit forming potential:** Low
Stimulation dosage (injection): 20 to 40 milligrams	**Estimated value U.S. (based on procedure):** $29 per gram

	(street value calculated at $5.00 per 60 milligram hit = $83.00 per gram.

Procedure A: Preparation of THIOESCALINE

Materials:

1. 12 grams of bromovanillin (see intermediate 0019. 3,4,5-TMB, procedure B, step 2)	14. 150 milliliters of a 5% hydrochloric acid solution
2. 11 milliliters of a 25% sodium hydroxide solution	15. 50 milliliters of 35 to 38% hydrochloric acid
3. 390 milliliters of methylene chloride	16. 63 milliliters of nitromethane
4. 11 grams of methyl iodide	17. 500 milligrams of anhydrous ammonium acetate
5. 575 milligrams of decyltriethylammonium chloride	18. 2.4 grams of lithium aluminum hydride
6. 45 grams of anhydrous magnesium sulfate	19. 108 milliliters of dry tetrahydrofuran
7. 160 milliliters of methyl alcohol	20. 1.6 milliliters of 98% sulfuric acid
8. 3.4 grams of cyclohexylamine	21. 114 milliliters of 99% isopropyl alcohol
9. 235 milliliters of diethyl ether	22. 90 milliliters of a 10% sodium hydroxide solution
10. 10 grams of anhydrous sodium sulfate	23. 120 milliliters of a 10% sulfuric acid solution
11. 1.6 grams of tert-buty lithium	24. 20 grams of sodium hydroxide
12. 15 milliliters of dry hexane	25. 10 grams of dry hydrogen chloride gas
13. 3.6 grams of diethyl disulfide	

Summary: THIOESCALINE is prepared in a four step process starting with the formation of 3-bromo-N-cyclohexyl-4,5-dimethoxybenzylidenimine. This intermediate is actually prepared from a pre-intermediate prepared by the reaction of bromovanillin with sodium hydroxide and methyl iodide in the presence of water and a small amount of catalyst. The reaction takes about 24 hours, and afterwards, the reaction mixture is treated with solvent, and the solvent then recovered and evaporated to recover a residue. This residue is then recrystallized from alcohol to afford crystals of the pre-intermediate of 3-bromo-4,5-dimethoxybenzaldehyde. This intermediate is then converted into the first primary intermediate (3-bromo-N-cyclohexyl-4,5-dimethoxybenzylidenimine) by reaction with cyclohexylamine under a free flame at 120 Celsius. After heating with the free flame, the resinous reaction mixture is dissolved into ether, washed, dried, and then evaporated to recover an oily residue of the desired 3-bromo-N-cyclohexyl-4,5-dimethoxybenzylidenimine. This intermediate is then converted into 3-ethylthio-4,5-dimethoxybenzaldehyde by reaction with butyl lithium at a temperature of –80 Celsius. After the initial cold reaction, the reaction mixture is treated with diethyl disulfide, and the dry ice/acetone bath is removed and the reaction mixture is allowed to warm to room temperature. The reaction mixture is then diluted with dilute acid, and the aqueous acidic phase is then separated, and refluxed. After the reflux period, the reaction mixture is cooled, and a yellow oil is then separated—leaving behind an aqueous phase. The yellow oil is then re-refluxed with additional acid, and the resulting refined yellow oil is then combined with the previous left behind aqueous phase. All combined phases are then extracted with solvent, and the solvent then evaporated in the usual manner to afford the desired residue containing the desired 3-ethylthio-4,5-dimethoxybenzaldehyde. The 3-ethylthio-4,5-dimethoxybenzaldehyde is then converted into 3-ethylthio-4,5-dimethoxy-beta-nitrostyrene by condensation with nitromethane in the usual manner. After the reaction, the excess nitromethane is removed, and the left over oily residue is then dissolved into alcohol, and the alcohol mixture then provoked to induce crystallization of the desired product. The recovered precipitated crystals are then recrystallized from fresh alcohol. The purified crystals are then finally converted into the desired product of THIOESCALINE by reaction of the nitro intermediate with lithium aluminum hydride in the usual manner. After the reduction of the nitro-intermediate with the lithium salt, the reaction mixture is treated in the usual manner, and then desired product residue recovered after removal of the extraction solvent. The recovered residue is then dissolved into alcohol, and the desired crystalline product is then recovered in the usual manner.

Hazards: Extinguish all flames before using diethyl ether, tetrahydrofuran, hexane, and nitromethane, all of which are highly flammable and can form explosive mixtures with air. Wear gloves and use caution when handling lithium aluminum hydride, and avoid contact with water. Use caution when handling methyl alcohol, and avoid contact with flames, sparks, electric discharges and other methods of ignition—as methyl alcohol burns with a colorless flame. Wear gloves when handling tert-buty lithium, and avoid contact with water and alcohols—as a violent reaction may result. Wear gloves when handling sodium hydroxide, hydrochloric acid, and sulfuric acid, as they can cause skin irritation.

Procedure:

Personnel notes for procedure A: Thioescaline

Step 1: Preparation of 3-bromo-N-cyclohexyl-4,5-dimethoxybenzylidenimine

Part A. Into a suitable flask or beaker (equipped with motorized stirrer or other means), place 12 grams of bromovanillin, followed by 11 milliliters of a 25% sodium hydroxide solution. Thereafter, gently stir the entire mixture until a heavy precipitate forms. When a precipitate forms, add in 25 milliliters of cold water, and then stir the entire mixture for about 10 minutes. Then add in 40 milliliters of methylene chloride, followed by 11 grams of methyl iodide, and then followed by 375 milligrams of decyltriethylammonium chloride catalyst. Thereafter, rapidly stir the entire reaction mixture for about 24 hours. After stirring for about 24 hours, allow the entire reaction mixture to stand for about 1 hour with no stirring, and then place the entire reaction mixture into a seperatory funnel, and recover the lower methylene chloride layer. Then quickly extract the upper aqueous layer with one 50-milliliter portion of methylene chloride, and after the exaction, collect the lower methylene chloride layer, and then combine it with the previous methylene chloride layer. Now, wash the combined methylene chloride portion with three 50-milliliter portions of cold water, and then dry the washed methylene chloride portion by adding to it, 15 grams of anhydrous magnesium sulfate. Note: after the washings, the methylene chloride will be the lower layer each time. After adding in the anhydrous magnesium sulfate, stir the entire mixture for about 10 minutes (to absorb moisture), and then filter-off the magnesium sulfate. Then place this dried methylene chloride portion into a distillation apparatus, and distill-off the methylene chloride at 40 Celsius. When no more methylene chloride passes over or is collected, stop the distillation process, and recover the left over remaining residue (after it has cooled). Finally, recrystallize this recovered left over residue from 75 milliliters of methyl alcohol, and after the recrystallization process, vacuum dry or air-dry the filtered-off crystals. The result will be about 4 grams of the pre-intermediate of 3-bromo-4,5-dimethoxybenzaldehyde.

Part B. Repeat the above procedure to produce a total of 8 grams of the 3-bromo-4,5-dimethoxybenzaldehyde. Then place 7.7 grams of the 3-bromo-4,5-dimethoxybenzaldehyde into a suitable sized beaker, and then add in 3.4 grams of cyclohexylamine. Thereafter, heat this mixture to 120 Celsius using a Bunsen burner until no more water vapor is given off. Note: to determine when no more water vapor is given off, place a watch glass or other piece of glass over the heated mixture, and when no more water vapor condenses on the glass, the reaction is done. When no more water vapor is given-off, stop the heating process and allow the reaction mixture to cool to room temperature. Thereafter, dissolve this resinous reaction mixture (composed of a resinous semi-solid or viscous residue) into 90 milliliter of diethyl ether, and then rapidly stir the entire mixture for about 30 minutes. Thereafter, filter this ether mixture to remove any insoluble impurities (if any), and then wash this ether mixture with three 50-milliliter portions of cold water. After each washing portion, the ether will be the upper layer each time. After the washing process, dry the collected ether layer by adding to it, 10 grams of anhydrous sodium sulfate, and then stir the entire mixture for about 10 minutes—thereafter, filter-off the sodium sulfate. Then place this dried ether mixture into a distillation apparatus, and distill-off the ether at 40 Celsius. When no more ether passes over or is collected, stop the distillation process, and recover the left over oily residue (after it has cooled). Then place this oily left over residue aside for step 2.

Step 2: Preparation of 3-ethylthio-4,5-dimethoxybenzaldehyde

Into a suitable flask or beaker (equipped with motorized stirrer or other string means, and thermometer), place 6.5 grams of the product obtained in step 1, part B, followed by 65 milliliters of dry diethyl ether. Thereafter, stir this entire mixture for about 30 minutes, and then filter the ether mixture to remove any potential insoluble impurities. Thereafter, place this filtered ether mixture into a dry ice/acetone bath, and chill to –80 Celsius. Thereafter, slowly add in, a butyl lithium solution prepared by adding and dissolving 1.6 grams of tert-buty lithium into 15 milliliters of dry hexane. Note: during the addition of the butyl lithium/hexane solution, moderately stir the reaction mixture and keep its temperature around –80 Celsius at all times. After the addition of the butyl lithium solution, continue to rapidly stir the reaction mixture for about 10 minutes until a white precipitate forms. Thereafter, add in 3.6 grams of diethyl disulfide, and after the addition of the diethyl disulfide, remove the dry ice bath, and allow the reaction mixture to warm to room temperature. Note: during this warming period, rapidly stir the reaction mixture. When the reaction mixture reaches room temperature, add in 150 milliliters of a 5% hydrochloric acid solution, and then stir the entire acidic reaction mixture for about 30 minutes. Then place the entire acidic reaction mixture into a seperatory funnel, and remove the lower aqueous acidic layer. Thereafter, place this lower aqueous acidic layer into a reflux apparatus, and reflux at 100 Celsius for about 30 minutes. Then remove the heat source, and allow the acidic aqueous layer to cool to room temperature. Thereafter, place this cooled acidic aqueous portion into a seperatory funnel, and allow it to stand for 1 hour, whereupon, remove the lower yellowish oil. Note: in some cases, the yellow oil may be the upper layer—the upper aqueous layer should be set-aside for a short while. Then place this recovered yellow oil into a clean beaker or flask, and then add in 25 milliliters of methyl alcohol, followed by 50 milliliters of 35 to 38% hydrochloric acid. Thereafter, place this acidic yellowish oil mixture into the same reflux apparatus (just previously used), and reflux the mixture at 100 Celsius for about 30 minutes. After 30 minutes, stop the reflux process, and allow the yellowish oil mixture to cool to room temperature. Thereafter, combine this cooled yellowish oil mixture with the upper aqueous layer (which was set-aside for just a short while), and then extract this combined yellowish oil and aqueous layer with two 50-milliliter portions of methylene chloride, and after the

extraction, combine all methylene chloride portions (if not already done so). Note: after each extraction, the methylene chloride will be the lower layer each time. Then dry this combined methylene chloride portion by adding to it, 15 grams of anhydrous magnesium sulfate, and then stir the entire methylene chloride portion for about 10 minutes—then filter-off the magnesium sulfate. Finally, place this filtered methylene chloride portion into a distillation apparatus, and distill-off the methylene chloride at 40 Celsius. When no more methylene chloride passes over or is collected, stop the distillation process, and recover the left over oily residue (after it has cooled), and then set it aside for step 3. Note: this recovered residue can be purified by vacuum distillation at 125 Celsius under a vacuum of 0.40 millimeters of mercury to obtain a colorless oil of the pure 3-ethylthio-4,5-dimethoxybenzaldehyde.

Step 3: Preparation of 3-ethylthio-4,5-dimethoxy-beta-nitrostyrene

Into a standard reflux apparatus, place 4 grams of the product obtained in step 2, followed by 63 milliliters of nitromethane, followed by 500 milligrams of anhydrous ammonium acetate. Then reflux this entire mixture for about 1 hour at 102 Celsius. After refluxing for about 1 hour, quickly remove the reflux condenser and replace it with a conventional cold-water condenser (fitted with receiver flaks), and then distill-off the excess nitromethane at 102 Celsius. When no more nitromethane passes over or is collected, stop the distillation process, and recover the left over remaining reddish oily residue (after it has cooled). Then dissolve this left over remaining residue into 10 milliliters of boiling methyl alcohol, and then stir the alcoholic mixture for about 5 minutes, and then quickly filter-off any insoluble materials. Then allow the filtered alcoholic mixture to cool to room temperature. Thereafter, place this cooled alcohol mixture into a dry ice bath, and then scratch the sides of the vessel (the flask or beaker containing the alcohol mixture in the dry ice bath) several times to induce formation of a seed crystal. Thereafter, allow the alcohol mixture to stand at dry ice temperatures for about 1 hour, and then remove the alcohol mixture from the dry ice bath, and allow it to stand for 1 hour. Then filter-off the precipitated crystals of the desired nitro intermediate, and then vacuum dry or air-dry them. Then recrystallize these dried crystals from 50 milliliters of methyl alcohol, and after the recrystallization process, vacuum dry or air-dry the filtered-off crystals.

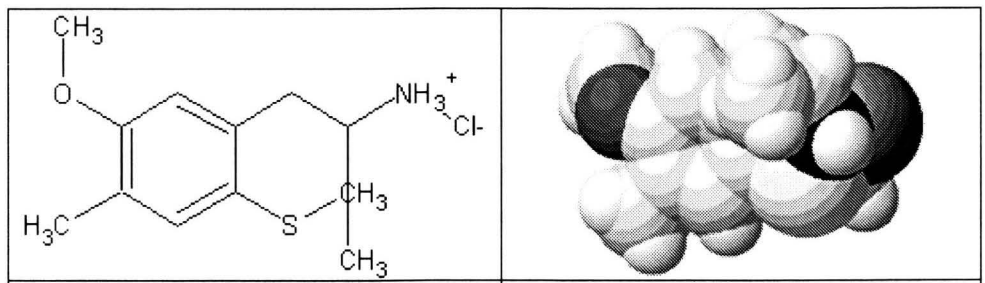
Step 4: Preparation of THIOESCALINE

Into a regular reflux apparatus (equipped with motorized stirrer or other stirring means, thermometer, and addition funnel), place 2.4 grams of lithium aluminum hydride, followed immediately by 90 milliliters of dry tetrahydrofuran, and then stir this mixture for about 10 minutes. Note: place a calcium chloride drying tube over the top of the reflux condenser to exclude moisture. Then place this mixture into an ice bath, and chill to about 0 Celsius. When its temperature reaches about 0 Celsius, slowly add in, in small portions, 1.6 milliliters of 98% sulfuric acid. Then place a solution, prepared by adding and dissolving 3.7 grams of the product obtained in step 3, into 18 milliliters of dry tetrahydrofuran, into the addition funnel. Thereafter, add this solution drop-wise, to the lithium aluminum hydride mixture over a period of about 15 minutes. During the addition, rapidly stir the lithium aluminum hydride mixture, and maintain its temperature below 5 Celsius at all times. After the addition, remove the ice bath, and allow the reaction mixture to warm to room temperature. Then reflux the entire reaction mixture at 100 Celsius for about 15 minutes. Afterwards, remove the heat source, and allow the reaction mixture to cool to room temperature. Then, place the entire reaction mixture into a clean beaker, and then add in 50 milliliters of 99% isopropyl alcohol (to destroy any excess lithium salts), and shortly after the addition of the alcohol, add in 90 milliliters of a 10% sodium hydroxide solution. After both additions, rapidly stir the entire reaction mixture for about 10 minutes. Then filter-off any insoluble impurities, and then wash these filtered-off impurities with two 25-milliliter portions of 99% isopropyl alcohol, and then combine both alcohol washing portions with the filtered reaction mixture. Then place the entire combined reaction mixture into a distillation apparatus, and distill the entire reaction mixture at 100 Celsius. When no more tetrahydrofuran, isopropyl alcohol, and water passes over or is collected, stop the distillation process, and recover the left over remaining resinous material (after it has cooled). Then dissolve this recovered left over resinous material into 120 milliliters of a 10% sulfuric acid solution, and then stir this mixture for about 10 minutes. Then filter-off any insoluble impurities, and then wash this filtered acidic mixture with four 50-milliter portions of methylene chloride. Note: after each washing portion, the acidic mixture will be the lower layer each time. After the washings, place the collected washed acidic mixture into a clean beaker, and then add in a sodium hydroxide solution prepared by adding and dissolving 20 grams of sodium hydroxide into 90 milliliters of cold water. Note: sodium hydroxide generates excessive heat when dissolved in water, so allow the alkaline solution to cool to room temperature before using. After adding in the sodium hydroxide solution, rapidly stir the entire alkaline mixture for about 10 minutes. Thereafter, extract this entire alkaline mixture with four 50-milliliter portions of methylene chloride, and after the exaction process, combine all methylene chloride portions (if not already done so), and then dry this combined methylene chloride portion by adding to it, 15 grams of anhydrous magnesium sulfate. Note: after each extraction portion, the methylene chloride will be the lower layer each time. After the addition of the magnesium sulfate, stir the entire methylene chloride portion for about 10 minutes, and then filter-off the magnesium sulfate. Them place this filtered methylene chloride portion into a distillation apparatus, and distill-off the methylene chloride at 40 Celsius. When no more methylene chloride passes over or is collected, stop the distillation process, and recover the left over remaining oily residue (after it has cooled). Now, dissolve this left over recovered oily residue into 14 milliliters of 99% isopropyl alcohol, and then stir this alcohol mixture for about 10 minutes. Thereafter, quickly filter this alcohol mixture to remove any insoluble impurities

(if any), and then place the filtered alcohol mixture into an ice bath, and chill to 0 Celsius. Finally, bubble into the alcohol mixture, 10 grams of dry hydrogen chloride gas (excess). After the addition of the hydrogen chloride gas, add in 30 milliliters of diethyl ether, and then allow the entire mixture to stand at 0 Celsius for about 1 hour. Thereafter, filter-off the precipitated crystals, wash them with two 25-milliliter portions of diethyl ether (several times using the same two 25-milliliter washing portions), and then vacuum dry or air-dry the washed crystals. The result will be about 3.3 grams of the desired product of THIOESCALINE.

Note: other salts of the freebase THIOESCALINE can be prepared in the usual manner, by replacing the hydrogen chloride with 98% sulfuric acid, pure tartaric acid, citric acid, or 80% phosphoric acid.

0032. 3M. 3M-Amphetamine. 5-Methoxy-4-methyl-2-methylthioamphetamine hydrochloride. *1-[5-methoxy-4-methyl-2-(methylthio)phenyl]propan-2-amine hydrochloride*



Structure | 3D model

3M forms the usual white to off-white crystals with a melting point of 196 Celsius. As usual, the impure crystals may have a slight pink to brownish color to them, with a melting point ranging from 194 to 198 Celsius. The crystals are insoluble in the usual organic solvents, but soluble in water and alcohol. 3M is similar to THIOMESCALINE, and it has been classified as a "happy" drug, which produces an excellent high upon ingestion. The high produced by 3M is the result of an extra ordinary array of effects that range from happiness, bursts of energy, feelings of well-being, and a overwhelming body feeling—the latter is similar to the "calming" or "satisfying" high produced by pain killers, but without the dizziness, drowsiness, or other side effects attributed to the use of pain killers. 3M also produces a superb psychological response in the brain leading to strong "positive" feelings towards the surrounding world. These positive feelings heavily contribute to the "happy" nature of this psychedelic amphetamine. Secondary effects of 3M include an increase in appetite, relaxing yet non-sedative like feelings, "stupor" or humorous responses to surrounding environment and related activities, enhancement to the taste of food, pleasant and enhancements to social situations (confidence and self-esteem towards the ability to socialize with others—especially perfect strangers), and enhancements to sight, memory, and mental awareness. Overall, 3M is an outstanding drug, which has mass potential for use as a "fun" drug for high school students and college students. 3M produces no side-effects, no withdrawal symptoms, and no cravings or addictive tendencies.

This substance is a controlled substance (psychedelic amphetamine) as listed in the US code of Federal regulations.

Toxicity: Low	**Rate of onset (average):** Moderate
Stimulation dosage (ingestion): 60 to 80 milligrams	**Duration of effects (average):** 8 to 10 hours
Stimulation dosage (inhalation): 35 to 45 milligrams	**Habit forming potential:** Very low
Stimulation dosage (injection): unknown—injection should be avoided	**Estimated value U.S. (based on procedure):** $31 per gram (street value calculated at $5.00 per 60 milligram hit = $83.00 per gram.

Procedure A: Preparation of 3M

Materials:

1. 13 grams of ortho-cresol	15. 2.9 grams of dichloromethyl methylether
2. 11 grams of dimethyl sulfoxide (DMSO)	16. 4 grams of anhydrous aluminum chloride
3. 190 milliliters of diethyl ether	17. 375 milliliters of a 5% sodium hydroxide solution
4. 8 milliliters of chlorosulfonic acid	18. 50 milliliters of cyclohexane
5. 90 milliliters of 99% isopropyl alcohol	19. 750 milligrams of lithium aluminum hydride
6. 50 milliliters of acetone	20. 110 milliliters of dry tetrahydrofuran
7. 640 milliliters of methylene chloride	21. Two drops of 98% sulfuric acid
8. 60 milliliters of a 5% sodium hydroxide solution	22. 128 milliliters of 99% isopropyl alcohol

9. 30 milliliters of 35 to 38% hydrochloric acid	23. 50 milliliters of a 25% sulfuric acid solution
10. 55 grams of anhydrous magnesium sulfate	24. 15 grams of sodium hydroxide
11. 135 milliliters of methyl alcohol	25. 10 grams of hydrogen chloride gas
12. 2 grams of potassium hydroxide	26. 37 milliliters of glacial acetic acid
13. 2 grams of methyl iodide	27. 1 gram of anhydrous ammonium acetate
14. 75 milliliters of a 25% sodium hydroxide solution	28. 3.9 grams of nitroethane

Summary: 3M is prepared in an exhaustive five step process starting with the formation of 2-methyl-4-(methylthio)phenol. This compound is prepared by reacting ortho-cresol with chlorosulfonic acid in the presence of DMSO and diethyl ether. The reaction is kept cool during the whole process, and afterwards, the insoluble solids are removed by filtration, washed with alcohol, and then dried. The dried crystals of the pre-intermediate dimethyl (4-hydroxy-3-methylphenyl)sulfonium chloride are then pyrolysis by heating with an open flame. During the heating process, a vigorous evolution of gas results, and after the gas evolution has ceased, the heating process is stopped, and the remaining residue is then dissolved into solvent, and the resulting solvent mixture is then extracted with base, treated with acid, and then extracted again into methylene chloride. The methylene chloride extract is then evaporated, and the resulting residue is then recrystallized from methyl alcohol. The resulting crystals of the desired 2-methyl-4-(methylthio)phenol are then converted into 2-methyl-4-(methylthio)anisole by reaction with methyl iodide in the presence of potassium hydroxide and methyl alcohol. The reaction is refluxed, and the desired product of 2-methyl-4-(methylthio)anisole is then collected by first, evaporation of the reaction mixture, followed by treating the left over residue with water and basifying the resulting aqueous mixture, and finally followed by extraction into methylene chloride. The methylene chloride is then removed in the usual manner, and the left over residue is then recovered. This left over residue containing predominantly the desired 2-methyl-4-(methylthio)anisole is then converted into 5-methoxy-4-methyl-2-(methylthio)benzaldehyde by reaction with dichloromethyl methyl ether in the presence of anhydrous aluminum chloride and ether. The reaction is generally mild, and after stirring for a short period of time, the reaction mixture is diluted with water, and then separated into two layers. The upper ether layer is set aside for a short while, and then combined with a methylene chloride extraction of the lower aqueous layer. The combined solvent portion is then washed, dried, and then evaporated to yield a residue. Thereafter, the residue is recrystallized from a suitable solvent to afford the desired product of 5-methoxy-4-methyl-2-(methylthio)benzaldehyde. This compound is then converted into 1-(5-methoxy-4-methyl-2-methylthiophenyl)-2-nitropropene by condensation with nitroethane in the usual manner. The reaction is refluxed for a awhile, and then allowed to stand overnight. The following day, the precipitated crystals of the nitro-intermediate are then filtered-off, and then recrystallized from methyl alcohol. The collected crystals of the nitro-intermediate are then converted into 3M by reaction with lithium aluminum hydride in the usual manner After the reaction, the reaction mixture is stripped of solvent, the left over residue is then dissolved into dilute acid, and the acidic mixture is then basified. The basified mixture is then extracted into chlorinated solvent, and this solvent is then evaporated. The left over residue is then dissolved into alcohol, treated with hydrogen chloride gas, and then diluted with ether to attribute precipitation of the desired product.

Hazards: Extinguish all flames before using diethyl ether, tetrahydrofuran, nitroethane, acetone, and cyclohexane, all of which are highly flammable and can form explosive mixtures with air. Wear gloves and use caution when handling lithium aluminum hydride, and avoid contact with water. Use caution when handling methyl alcohol, and avoid contact with flames, sparks, electric discharges and other methods of ignition—as methyl alcohol burns with a colorless flame. Wear gloves when handling sodium hydroxide, potassium hydroxide, hydrochloric acid, glacial acetic acid, and sulfuric acid, as they can cause skin irritation. Wear gloves when handling dimethyl sulfoxide and use proper ventilation—DMSO is toxic and can be absorbed through the skin. Wear gloves and use proper ventilation when handling chlorosulfonic acid, which is irritating to the eyes, nose, and throat, and in which can cause skin irritation. Use care when handling anhydrous aluminum chloride, and avoid contact with water. Dichloromethyl methylether should be handled with good ventilation, and inhalation and skin contact of the substance should be avoided. Methyl iodide should be stored in amber glass bottles in a cool dry place, and avoid contact with sunlight.

Procedure:

Personnel notes for procedure A: 3M

Step 1: Preparation of 2-methyl-4-(methylthio)phenol

Into a suitable beaker or flask (equipped with motorized stirrer or other stirring means), place 13 grams of ortho-cresol, followed by 11 grams of dimethyl sulfoxide (DMSO), followed by 60 milliliters of diethyl ether. Then stir the entire mixture

for about 10 minutes, and then place the flask or beaker into an ice bath, and chill to 0 Celsius. When the temperature of the mixture reaches about 0 Celsius, slowly add in, 8 milliliters of chlorosulfonic acid, drop-wise, over a period of about 15 minutes, while rapidly stirring the ortho-cresol mixture and maintaining its temperature below 5 Celsius at all times. During the addition of the chlorosulfonic acid, rapidly stir the ortho-cresol mixture. After the addition of the chlorosulfonic acid, remove the ice bath and allow the reaction mixture to warm to room temperature. During this warming period, rapidly stir the reaction mixture. When the temperature of the reaction mixture reaches room temperature, rapidly stir the entire reaction mixture for about 2 ½ hours. After 2 ½ hours, filter-off the insoluble solids, and then place these filtered-off solids into 60 milliliters of 99% isopropyl alcohol, and then rapidly stir this alcoholic mixture for about 30 minutes at room temperature. Thereafter, filter-off the pink insoluble solids (which will be suspended in the alcohol), and then wash these filtered-off solids with 30 milliliters of fresh 99% isopropyl alcohol, and then vacuum dry or air-dry the washed solids. Then recrystallize these dried solids from 100 milliliters of a solvent mixture prepared by adding 50 milliliters of acetone to 50 milliliters of water, and after the recrystallization process, vacuum dry or air-dry the filtered-off crystals. Now, place these recrystallized dried crystals into a suitable sized beaker, and then gently heat these crystals at 156 Celsius using a Bunsen burner. Note: while heating the crystals at 156 Celsius, an evolution of methyl chloride gas will result. Continue to heat the crystals at 156 Celsius until no more gas is evolved. When no more gas is evolved, stop the heating process, and allow the heated mixture to cool to room temperature. Thereafter, dissolve this cooled mixture (residue) into 40 milliliters of methylene chloride, and then stir the mixture for about 10 minutes. Thereafter, filter-off any insoluble materials (if any), and then extract this filtered methylene chloride mixture with three 20-milliliter portions of a 5% sodium hydroxide solution. After the extraction, combine all dilute sodium hydroxide portions, if not already done so, and then acidify this combined dilute sodium hydroxide portion by adding to it, 30 milliliters of 35 to 38% hydrochloric acid. Note: after each extraction, the dilute sodium hydroxide will be the upper layer each time. After adding in the concentrated hydrochloric acid, rapidly stir the now acidic mixture for about 30 minutes. Thereafter, extract this combined acidic portion with three 50-milliliter portions of methylene chloride, and after the extraction, combine all methylene chloride portions (if not already done so), and then dry this combined methylene chloride portion, by adding to it, 15 grams of anhydrous magnesium sulfate. Note: after each methylene chloride extraction, the methylene chloride will be the lower each time. After adding in the anhydrous magnesium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Finally, place this filtered methylene chloride portion into a distillation apparatus, and distill-off the methylene chloride at 40 Celsius. When no more methylene chloride passes over or is collected, stop the distillation process, and recover the left over remaining residue (after it has cooled). Then recrystallize this left over recovered residue from 75 milliliters of methyl alcohol, and after the recrystallization process, vacuum dry or air-dry the filtered-off crystals. The result will be about 4 grams of the desired product with a melting point ranging from 35 to 40 Celsius.

Step 2: Preparation of 2-methyl-4-(methylthio)anisole

Into a standard reflux apparatus, equipped with motorized stirrer or other stirring means, thermometer, and addition funnel, place 4 grams of the product obtained in step 1, followed by 20 milliliters of methyl alcohol. Thereafter, stir the entire mixture briefly to form a uniform mix. Thereafter, place into the addition funnel, a solution prepared by adding 2 grams of potassium hydroxide to 10 milliliters of methyl alcohol. Note: adding sodium hydroxide to methyl alcohol generates some heat—never mind this and go ahead and add this alkaline alcohol mixture to the addition funnel. Thereafter, add this alkaline alcoholic mixture to the reaction mixture drop-wise, over a period of only about 5 to 10 minutes. During the addition, rapidly stir the reaction mixture. Immediately after the addition, add in, in one portion, 2 grams of methyl iodide. After this addition, reflux the entire reaction mixture at 68 Celsius for about 3 hours. After refluxing the reaction mixture for about 3 hours, quickly replace the reflux condenser with a conventional cold-water condenser (fitted with a receiver flask), and then distill the reaction mixture at 68 Celsius to remove the methyl alcohol. When no more methyl alcohol passes over or is collected, stop the distillation process, and recover the left over remaining residue (after it has cooled). Then place this recovered left over residue into a clean beaker, and then add in 75 milliliters of water. Thereafter, stir this aqueous mixture for about 30 minutes, and then add in, 75 milliliters of a 25% sodium hydroxide solution, and then stir the entire alkaline mixture for about 10 minutes. After 10 minutes, extract this entire alkaline mixture with three 50-millilter portions of methylene chloride, and after the extraction process, combine all methylene chloride portions (if not already done so), and then dry this combined methylene chloride portion, by adding to it, 15 grams of anhydrous magnesium sulfate. Note: after each extraction, the methylene chloride will be the lower layer each time. After adding in the anhydrous magnesium sulfate, stir the entire combined methylene chloride portion for about 10 minutes, and then filter-off the magnesium sulfate. Finally, place this filtered methylene chloride portion into a distillation apparatus, and distill-off the methylene chloride at 40 Celsius. When no more methylene chloride passes over or is collected, stop the distillation process, and recover the left over remaining residue (after it has cooled), and then set this residue aside for step 3.

Step 3: Preparation of 5-methoxy-4-methyl-2-(methylthio)benzaldehyde

Into a suitable beaker or flask (equipped with motorized stirrer or other stirring means), place 3.6 grams of the product obtained in step 2, followed by 2.9 grams of dichloromethyl methylether, and then followed by 100 milliliters of diethyl ether. Thereafter, stir this entire mixture for about 10 minutes to form a uniform mix. Then add in, in small portions, 4 grams of

anhydrous aluminum chloride over a period of about 1 minute. During the addition, rapidly stir the reaction mixture, and after the addition, continue to stir the reaction mixture for about 20 minutes. After 20 minutes, the reaction mixture should have a dark red appearance to it. After 20 minutes, slowly and carefully add in, 90 milliliter of cold water (to destroy the aluminum chloride), and then stir the entire diluted reaction mixture for about 1 hour—if after stirring for 1 hour, there are any insoluble yellow solids, continue to stir the reaction mixture until the yellow solids dissolve. After stirring for about 1 hour, place the entire reaction mixture (which will be a two-phase mixture) into a seperatory funnel, and collect the upper ether layer. Once the upper ether layer has been collected, set it aside for just a moment. Then, to the lower aqueous layer, extract this aqueous layer with three 50-milliliter portions of methylene chloride. After the extraction process, combine all methylene chloride portions (if not already done so), and then combine this combined methylene chloride portion with the previous collected ether layer. Note: after each extraction, the methylene chloride will be the lower layer each time. Thereafter, wash this combined solvent portion with three 75-milliliter portions of a 5% sodium hydroxide solution. Note: after each washing portion, the combined solvent portion will be the lower layer each time. After the washing portion, dry this combined washed solvent portion by adding to it, 10 grams of anhydrous magnesium sulfate, and then stir the entire solvent mixture for about 10 minutes—then filter-off the magnesium sulfate. Finally, place this combined solvent portion into a distillation apparatus, and distill-off the ether and methylene chloride at 40 Celsius. When no more ether or methylene chloride passes over or is collected, stop the distillation process, and recover the left over remaining residue (after it has cooled). Thereafter, mix this collected residue with 50 milliliters of cyclohexane, and then stir the entire mixture for about 30 minutes. Thereafter, filter-off any insoluble solids, and then recrystallize the desired product from this filtered cyclohexane mixture. After the recrystallization process, vacuum dry or air-dry the collected crystals of the desired product. Note: during the recrystallization process, only evaporate-off about 60 to 70% of the cyclohexane—as the remaining cyclohexane (after concentration) will contain a dissolved by-product.

Step 4: Preparation of 1-(5-methoxy-4-methyl-2-methylthiophenyl)-2-nitropropene

Into a standard reflux apparatus (equipped with motorized stirrer or other stiring means), place 12 milliliters of glacial acetic acid, followed by 3 grams of the product obtained in step 3, followed by 1 gram of anhydrous ammonium acetate, and then followed by 2.4 grams of nitroethane. Thereafter, reflux the entire mixture for about 8 hours at 115 Celsius, and during the reflux period, moderately stir the reaction mixture. After refluxing for about 8 hours, quickly add in 1.5 grams of additional nitroethane (through the top of the reflux condenser), and then continue refluxing and stirring the reaction mixture for about 8 additional hours. Thereafter, remove the heat source and allow the reaction mixture to cool to room temperature. Then allow the entire reaction mixture to stand at room temperature overnight (without stirring). The next day, filter-off the precipitated orange crystals, wash them with one 25-milliliter portion of cold glacial acetic acid (several times using the same washing portion), and then vacuum dry or air-dry the crystals. Finally, recrystallize the dried crystals from 30 milliliters of boiling methyl alcohol. After the recrystallization process, vacuum dry or air-dry the filtered-off crystals. The result will be about 2 grams of the desired product with a melting point of 84 Celsius

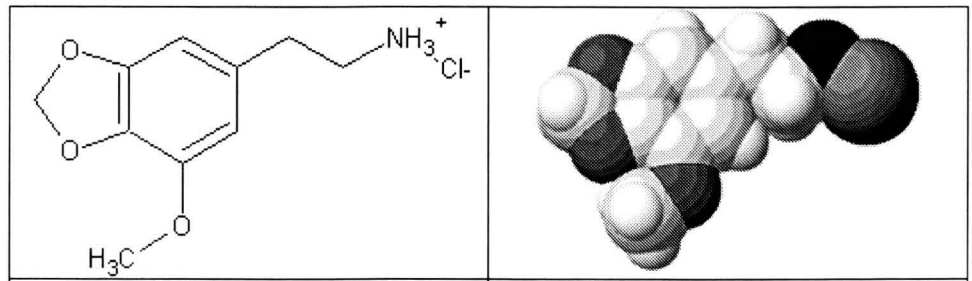
Step 5: Preparation of 5-methoxy-4-methyl-2-methylthioamphetamine (3M)

Into a suitable reflux apparatus, equipped with motorized stirrer or other stirring means, and addition funnel, place 750 milligrams of lithium aluminum hydride, followed by 40 milliliters of dry tetrahydrofuran. Note: place a calcium chloride drying tube onto the top of the reflux condenser. Thereafter place the lithium aluminum hydride mixture into an ice bath and chill to about 0 Celsius. Then place two drops of 98% sulfuric acid into the lithium aluminum hydride mixture, and then briefly stir the entire mixture for about 10 minutes at 0 Celsius. Note: during the addition of the sulfuric acid, stir the lithium aluminum hydride mixture and maintain its temperature below 5 Celsius at all times. Note: the few drops of sulfuric acid can be added through the top of the reflux condenser (by temporarily removing the calcium chloride drying tube). After the addition of the sulfuric acid, prepare a solution by adding and dissolving 1.5 grams of the product obtained in step 4 into 20 milliliters of dry tetrahydrofuran—then place this solution into the addition funnel. Then add drop-wise, this solution (which is in the addition funnel), to the lithium aluminum hydride mixture over a period of about 5 minutes, while rapidly stirring the reaction mixture and maintaining its temperature below 5 Celsius at all times. After the addition, continue to stir the reaction mixture for an additional 10 minutes, and then reflux the reaction mixture gently, at 68 Celsius for about 30 minutes. After refluxing for about 30 minutes, remove the heat source and allow the reaction mixture to cool to room temperature. Thereafter, pour the entire reaction mixture into a suitable sized beaker, and then place this beaker into an ice bath, and chill to about 0 Celsius. Then slowly and carefully add in, 50 milliliters of 99% isopropyl alcohol, and then stir the entire reaction mixture for about 10 minutes. Afterwards, add in 150 milliliters of a 5% sodium hydroxide solution, and then stir the entire alkaline reaction mixture for about 30 minutes. Then filter the alkaline reaction mixture to remove any insoluble solids, and then briefly wash these filtered-off solids with two 25-milliliter portions of tetrahydrofuran, followed by washing with two 25-milliliter portions of 99% isopropyl alcohol. After the washings, add both the tetrahydrofuran and isopropyl alcohol washing portions to the filtered reaction mixture, and then place this combined reaction mixture into a distillation apparatus, and distill-off the tetrahydrofuran and isopropyl alcohol at 85 Celsius. When no more tetrahydrofuran and isopropyl alcohol passes over or is collected, stop the distillation process, and recover the left over remaining oily residue (after it has cooled). Thereafter, dissolve this recovered oily residue into 50 milliliters of a 25% sulfuric acid solution, and then stir the entire aqueous acidic mixture for

about 10 minutes. Thereafter, wash this aqueous acidic mixture with two 25-millliter portions of methylene chloride. Note: after each washing, the aqueous acidic portion will be the lower layer each time—the methylene chloride can be recycled or discarded if desired. After the washing process, place the aqueous acidic portion into a clean beaker, and then add in a sodium hydroxide solution prepared by adding and dissolving 15 grams of sodium hydroxide into 100 milliliters of cold water. Note: sodium hydroxide generates excessive heat when dissolved in water so allow the alkaline mixture to cool before using. After the addition of the sodium hydroxide solution, rapidly stir the now alkaline mixture for about 30 minutes. Then extract this alkaline mixture with two 50-milliliter portions of methylene chloride. After the extraction process, combine all methylene chloride portions (if not already done so), and then dry this combined methylene chloride portion by adding to it, 15 grams of anhydrous magnesium sulfate. Note: after each extraction, the methylene chloride will be the lower layer each time. After the addition of the sodium hydroxide solution, stir the entire methylene chloride portion for about 10 minutes, and then filter-off the magnesium sulfate. Finally, place this filtered methylene chloride portion into a distillation apparatus, and remove the methylene chloride at 40 Celsius. When no more methylene chloride passes over or is collected, stop the distillation process, and recover the left over remaining residue (after it has cooled). Then mix this recovered residue with 8 milliliters of 99% isopropyl alcohol, and then stir the entire mixture for about 10 minutes. Then quickly filter the mixture to remove any potential insoluble impurities, and then place this filtered isopropyl alcohol mixture into a suitable size beaker, and then add in 10 milliliters of additional 99% isopropyl alcohol. Then bubble into this alcohol mixture, 10 grams of hydrogen chloride gas. After the addition of the hydrogen chloride gas, add in 10 milliliters of additional 99% isopropyl alcohol, and thereafter, add in 30 milliliters of diethyl ether. Then place this solvent mixture into an ice bath, and chill to about 0 Celsius. Then allow this solvent mixture to stand for 1 hour at 0 Celsius, and thereafter, filter-off the precipitated crystals of the desired 3M, and then vacuum dry or air-dry the crystals.

Note: other salts of the freebase 3M can be prepared in the usual manner, by replacing the hydrogen chloride with 98% sulfuric acid, pure tartaric acid, citric acid, or 80% phosphoric acid.

0033. LOPHOPHINE. 3-Methoxy-4,5-methylenephenethylamine hydrochloride. *2-(7-methoxy-1,3-benzodioxol-5-yl)ethanamine hydrochloride*



Structure	3D model

LOPHOPHINE forms colorless to whitish, to off-white crystals with melting point ranging from 160 to 164 Celsius. The crystals are soluble in water, alcohol, and hot acetonitrile, but insoluble in ether, and methylene chloride. LOPHOPHINE is related to MESCALINE, and may be found in peyote in small amounts. It is a psychologically active derivative of MESCALINE with similar properties, but with fewer psychedelic enhancements as found in the predecessor, MESCALINE. LOPHOPHINE produces superb, relaxing, and good natured mood elevations with enhancements to feelings such as feelings of well-being, self confidence, calming and relaxing natured moods, and a series of mild psychedelic natured alterations ranging from mild sight enhancements, slight color elevations, and a slight increase in depth perception—however, all psychedelic enhancements are mild, and in general, are considered to be far less then that of MESCALINE. Overall, LOPHOPHINE is a good-natured drug, that produces mild psychedelic effects, but with good euphoric feelings and feelings of well-being.

This substance is a controlled substance (psychedelic amphetamine) as listed in the US code of Federal regulations.

Toxicity: Low	**Rate of onset (average):** Slow (may take up to 2 hours for effects to be realized, but some cases have reported effects beginning at the 1 hour mark).
Stimulation dosage (ingestion): 150 to 250 milligrams	**Duration of effects (average):** 5 hours
Stimulation dosage (inhalation): unknown	**Habit forming potential:** Low
Stimulation dosage (injection): unknown	**Estimated value U.S. (based on procedure):** $28 per gram

Procedure A: Preparation of LOPHOPHINE

Materials:

1. 150 milliliters of dimethylformamide (DMF)	10. 3.4 grams of anhydrous ammonium acetate
2. 8.4 grams of 5-hydroxyvanillin (obtained in step 3 of procedure B of Intermediate-0019. 3,4,5-TMB)	11. 3.5 grams of lithium aluminum hydride
3. 29 grams of potassium fluoride	12. 276 grams of a pre-chilled 7% sulfuric acid solution
4. 154.6 grams of methylene chloride	13. 89 grams of potassium sodium bitartrate
5. 825 milliliters of diethyl ether	14. 30 grams of sodium hydroxide
6. 225 milliliters of a 10% sodium carbonate solution	15. 15 grams of anhydrous magnesium sulfate
7. 15 grams of anhydrous sodium sulfate	16. 10 grams of hydrogen chloride gas
8. 100 milliliters of dry hexane	17. 150 milliliters of boiling acetonitrile
9. 90 milliliters of glacial acetic acid	18. 6.6 milliliters of nitromethane

Summary: LOPHOPHINE is prepared in a three-step process starting with the formation of myristicinaldehyde. This intermediate is prepared by reacting 5-hydroxyvanillin with potassium fluoride in the presence of methylene chloride under reflux. After the reaction, the reaction mixture is cooled, extracted with ether, and the resulting ether extracts are then treated in the usual manner. Upon evaporation of the ether, followed by recrystallization, the desired myristicinaldehyde is obtained. The myristicinaldehyde is then converted into the nitro styrene intermediate by condensation with nitromethane in glacial acetic acid. The reaction mixture is then refluxed, and then allowed to stand at ice bath temperatures. The precipitated crystals of the nitro styrene intermediate are then filtered-off, washed, and then dried. The dried yellow nitro styrene crystals can then be converted into the desired LOPHOPHINE by reaction with lithium aluminum hydride in the usual manner. The desired product is then collected by extraction into methylene chloride only after treating the reaction mixture with a series of reagents including water, acid, and base. The solvent extract containing the desired product is then evaporated, and the left over residue is then dissolved into ether, and the product is then precipitated by the addition of hydrogen chloride gas. The precipitated LOPHOPHINE product is then collected by filtration, washed, and then recrystallized from boiling acetonitrile.

Hazards: Extinguish all flames before using diethyl ether, nitromethane, and hexane, all of which are highly flammable and can form explosive mixtures with air. Wear gloves and use caution when handling lithium aluminum hydride, and avoid contact with water. Wear gloves when handling sodium hydroxide, hydrogen chloride gas, glacial acetic acid, and sulfuric acid, as they can cause skin irritation—hydrogen chloride gas is an irritating gas so avoid inhalation. Wear gloves when handling acetonitrile, and use proper ventilation—acetonitrile is toxic and can be absorbed through the skin.

Procedure:

Personnel notes for procedure A: Lophophine

Step 1: Preparation of Myristicinaldehyde

Into a suitable flask or beaker, equipped with motorized stirrer or magnetic stirrer, place 150 milliliters of dimethylformamide (DMF), followed by 8.4 grams of 5-hydroxyvanillin (obtained in step 3 of procedure B of Intermediate-0019. 3,4,5-TMB). Then briefly stir the entire mixture to dissolve all solids. Thereafter, gradually add in 29 grams of potassium fluoride, and then rapidly stir the entire reaction mixture for about 1 hour. Note: if after 1 hour, the reaction mixture is still warm, continue to stir the reaction mixture until it cools to room temperature. Thereafter, add in 4.6 grams of methylene chloride, and then place the entire reaction mixture into a reflux apparatus, and reflux at 120 Celsius for 1 hour. After refluxing the reaction mixture at 120 Celsius for 1 hour, remove the heat source, and allow the reaction mixture to cool to room temperature. Now, extract the entire reaction mixture with three 50-milliliter portions of diethyl ether, and after the extraction period, combine all ether extracts (if not already done so), and then wash this combined ether portion with three 25-milliliter portions of cold water. Note: after the extraction and washing portions, the ether will be the upper layer each time. After the extraction and water washing portions, wash the ether portion with three 75-milliliter portions of a 10% sodium carbonate solution (based by weight on the anhydrous sodium carbonate in water, not any of the hydrates). After each washing portion, the ether will be the upper layer each time. After the washing of the ether portion with sodium carbonate solution, dry the ether portion by adding to it, 15 grams of anhydrous sodium sulfate, and then stir the entire mixture for about 10 minutes. Finally, filter-off the sodium sulfate, and then place the filtered ether portion into a distillation apparatus, and distill-off the diethyl ether. After the diethyl ether has been completely removed by distillation, recover the left over remaining residue (after it has cooled), and then recrystallize it from 100 milliliters of dry hexane. After the recrystallization process, vacuum dry or air-dry the filtered-off crystals.

Step 2: Preparation of the nitro styrene intermediate

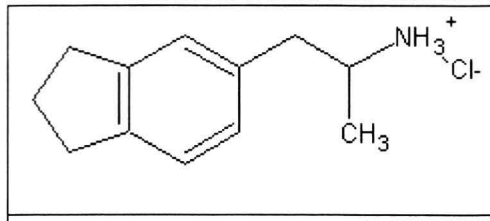
Into a standard reflux apparatus, equipped with motorized stirrer or other stirring means, place 10 grams of myristicinaldehyde (prepared in step 1), followed by 40 milliliters of glacial acetic acid. Thereafter, briefly stir the mixture to form a uniform mix, and then add in 6.6 milliliters of nitromethane, followed by 3.4 grams of anhydrous ammonium acetate. Then reflux this entire mixture for about 1 hour at 102 Celsius with moderate stirring. After the refluxing period, remove the heat source, and allow the reaction mixture to cool to room temperature. Thereafter, pour the entire reaction mixture into a suitable sized beaker, and then place this beaker into an ice bath, and chill to about 0 Celsius. Then add in 50 milliliters of ice-cold water, and then stir the entire mixture for about 1 hour at a temperature below 10 Celsius. After 1 hour, stop the stirring process, and allow the entire reaction mixture to stand in the ice bath for about 1 hour. Thereafter, filter-off the precipitated yellow crystals, wash the crystals with two 25-millilter portions of cold glacial acetic acid, and then vacuum dry or air-dry the crystals. The result will be about 3.8 grams of the desired nitro styrene product. Note: the filtered reaction mixture can be extracted with methylene chloride (three 50-millilter portions), and the combined methylene chloride portion then washed with several 50-millilter portions of a 5% sodium hydroxide solution, and the resulting washed methylene chloride portion then extracted to yield a dark residue. This dark residue can be recycled by mixing with additional glacial acetic acid, and nitromethane as in the process described in this step, for a second crop of yellow nitro styrene product.

Step 3: Preparation of LOPHOPHINE

Into a standard reflux apparatus, equipped with motorized stirrer or other stirring means, and addition funnel, place 3.5 grams of lithium aluminum hydride, followed by 220 milliliters of dry diethyl ether. Thereafter, gently reflux this mixture at 68 Celsius, and when the mixture begins to reflux, place a solution (into the addition funnel) prepared by adding and dissolving 3.8 grams of the product obtained in step 2, into 200 milliliters of dry diethyl ether. Thereafter, slowly add drop-wise, this solution, to the lithium aluminum hydride mixture over a period of about 5 to 6 hours. During the addition, rapidly stir the reaction mixture and maintain its temperature below 70 Celsius at all times. After the addition, continue to rapidly stir the reaction mixture, and maintain the reflux at 68 Celsius for about 28 hours. After 28 hours, remove the heat source, and allow the reaction mixture to cool to room temperature. Then pour the entire reaction mixture into a clean beaker, and then place this beaker into an ice bath, and chill to about 0 Celsius. Thereafter, slowly and carefully add in, 276 grams of a pre-chilled 7% sulfuric acid solution. During the addition of the acid, rapidly stir the reaction mixture. After the addition of the cold dilute sulfuric acid solution, place the entire diluted acidic reaction mixture into a seperatory funnel, and remove the lower acidic aqueous layer. Note: the upper ether layer can be recycled if desired. Thereafter, wash this lower acidic aqueous layer with two 40-millilter portions of fresh cold diethyl ether. Note: after each washing portion, the dilute acidic aqueous portion, will be the bottom layer each time—the upper ether layers can be recycled if desired. Now, place this collected washed dilute acidic aqueous layer into a suitable sized beaker, and then add in 89 grams of potassium sodium bitartrate, and then rapidly blend the entire mixture for about 30 minutes. Thereafter, slowly add in 30 grams of sodium hydroxide, in very small portions. Note: during the addition of the sodium hydroxide rapidly stir the mixture, and after the addition, rapidly stir the mixture for about 1 hour. Then, extract this entire alkaline mixture with three 50-milliliter portions of methylene chloride, and after the extraction process, combine all methylene chloride portions (if not already done so), and then dry this combined methylene chloride portion by adding to it, 15 grams of anhydrous magnesium sulfate. Note: after each extraction portion, the methylene chloride will be the lower layer each time. After adding in the anhydrous magnesium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Finally, place this dried filtered methylene chloride portion into a distillation apparatus, and distill-off the methylene chloride at 40 Celsius. When no more methylene chloride passes over or is collected, stop the distillation process, and collect the left over remaining residue (after it has cooled). Then dissolve this recovered left over residue into 75 milliliters of diethyl ether, and then stir the entire mixture for about 30 minutes—thereafter, quickly filter the mixture to remove any insoluble materials. Then place this filtered ether mixture into a suitable sized beaker, and then bubble into this ether mixture, 10 grams of hydrogen chloride gas (excess). After the addition of the hydrogen chloride gas, allow the ether mixture to stand for about 1 hour, and then filter-off the precipitated solids. Then wash these filtered-off solids with two 25-milliliter portions of diethyl ether, and then vacuum dry or air-dry the solids. Now, recrystallize these dried solids from 150 milliliters of boiling acetonitrile, and after the recrystallization process, wash the filtered-off collected crystals with two 25-milliter portions of cold diethyl ether, and then vacuum dry or air-dry the filtered-off crystals.

Note: other salts of the freebase LOPHOPHINE can be prepared in the usual manner, by replacing the hydrogen chloride gas with 98% sulfuric acid, pure tartaric acid, citric acid, or 80% phosphoric acid.

0034. IAP. Indanylamphetamine hydrochloride. *1-(2,3-dihydro-1H-inden-5-yl)propan-2-amine hydrochloride*



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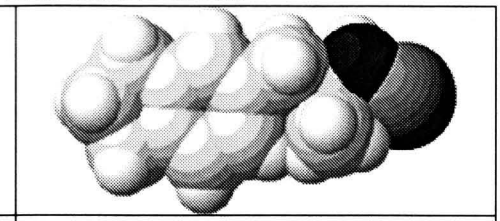


3D model

IAP forms colorless to white, to amber, to brown crystals (depending on purity), with a melting point ranging from 215 to 220 Celsius. The pure crystals have a melting point of about 219 Celsius. IAP is related to ecstasy and MDA in the areas of psychological activity, but its exact psychological nature has yet to be fully realized. As of so far, IAP is classified as an experimental psychedelic amphetamine with little or no stimulant action. Reports have shown that the use of IAP leads to a psychological high resembling MDA in nature, with the usual feelings of well being, and other good-natured feelings. IAP is a very potent substance, and its use should be taken with care and caution. Human testing should only be carried out by trained and professional medical personnel only. Misuse of this substance may lead to death, or other less severe medical problems. Overall, the use of IAP is by no means a threat to life, but its experimental label dictates that its use should be monitored, and the dosage should be kept at a minimum until it is other wise shown that it can be increased. In essence, this compound has excellent potential for use in "feel good" drugs, either by itself, or when combined with ecstasy—the latter has demonstrated to be of high concern, as mixtures of IAP and ecstasy can produce outstanding and very good effects upon the body. IAP mixed with methamphetamine, LSD, heroin, cocaine, and many other drugs may actually increase the potency and "high" effect of the drug up to three times the normal level—however, this should be taken under caution as increased potency may lead to increased over doses in customers.

This substance is a controlled substance (psychedelic amphetamine) as listed in the US code of Federal regulations.

Toxicity: Unknown—experimental results show toxicity is High	**Rate of onset (average):** Unknown
Stimulation dosage (ingestion): 20 to 40 milligrams	**Duration of effects (average):** Unknown—may last up to 10 hours
Stimulation dosage (inhalation): unknown	**Habit forming potential:** Unknown—has been estimated to be low
Stimulation dosage (injection): 2 to 5 milligrams	**Estimated value U.S. (based on procedure):** Unknown (street value could be $4.00 per 40 milligram hit, equaling $100 per gram

Procedure A: Preparation of IAP

Materials:

1. 12 grams of indan	10. 3.6 grams of anhydrous ammonium acetate
2. 570 milliliters of methylene chloride	11. 100 milliliters of methyl alcohol
3. 18 milliliters of 99% tin-IV-chloride	12. 1000 milliliters of dry tetrahydrofuran
4. 12 grams of dichloromethyl methyl ether	13. 6 grams of lithium aluminum hydride
5. 150 milliliters of a 5% hydrochloric acid solution	14. 530 milliliters of diethyl ether
6. 100 milliliters of a 23% sodium chloride solution	15. 500 milliliters of a 7% hydrochloric acid solution
7. 10 grams of powdered charcoal	16. 45 grams of sodium hydroxide
8. 40 grams of anhydrous magnesium sulfate	17. 15 grams of hydrogen chloride gas
9. 90 milliliters of nitroethane	

Summary: Indanylamphetamine hydrochloride can be prepared in a rather simple three-step process starting with the formation of indan-5-aldehyde. This intermediate is readily prepared by treating indane with dichloromethyl methyl ether in the presence of tin-IV-chloride and methylene chloride. The reaction is kept at ice-cold temperatures, and then quenched with ice water. The diluted reaction mixture is then separated, and the lower organic phase is washed, dried, and then removed to recover the left over oily residue. This oily residue is then converted into the nitro intermediate by condensation of the indan aldehyde with nitroethane in the presence of a small amount of catalyst. The reaction is refluxed for about 8 hours, and then cooled. The cooled mixture is then chilled, and any precipitated crystals are then filtered-off. The filtered reaction mixture is then carefully distilled (to remove any excess nitroethane), and the left over remaining residue is then combined with any previously filtered-off crystals, and the combined residue is then extracted into methylene chloride. The methylene chloride is dried, and then evaporated to recover the left over oily residue, which is then fractionally recrystallized from methyl alcohol. The collected crystals of the nitro intermediate are then converted into the desired indanylamphetamine hydrochloride by reduction with lithium aluminum hydride in the usual manner. After the reduction, the reaction mixture is diluted with water,

and then distilled to remove all liquids. When nothing but left over residue remains, the residue is taken up into solvent, extracted with acid, and the resulting aqueous acidic portion is then separated, extracted with base, and then taken up into methylene chloride. The methylene chloride is then evaporated, and the left over residue is then dissolved into ether. The desired product is then precipitated by the addition of hydrogen chloride, in the usual manner.

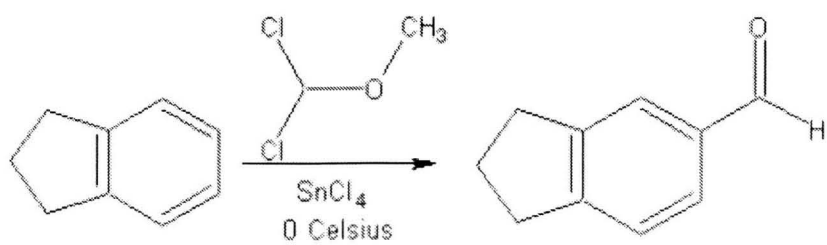
Hazards: Extinguish all flames before using diethyl ether, tetrahydrofuran, and nitroethane, both of which are highly flammable and can form explosive mixtures with air. Wear gloves and use caution when handling lithium aluminum hydride, and avoid contact with water. Wear gloves when handling sodium hydroxide, hydrochloric acid, and hydrogen chloride gas, as they can cause skin irritation—hydrogen chloride gas is an irritating gas so avoid inhalation. Wear gloves when handling dichloromethyl methyl ether, and avoid inhalation and skin contact. Wear gloves when handling tin-IV-chloride, and avoid contact with water and moisture. Powdered charcoal may readily ignite upon exposure to ignition, so keep all sources of ignition away. Methyl alcohol burns with a colorless flame, so extinguish all sources of ignition before using—as burning methanol cannot be seen, and hence, gives no warning of its presence.

Procedure:

Personnel notes for procedure A: IAP

Step 1: Preparation of Indan-5-aldehyde

Into a suitable flask or beaker (equipped with motorized stirrer or other stirring means), place 12 grams of indan, followed by 120 milliliters of methylene chloride. Thereafter, place this flask or beaker into an ice bath, and chill to about 0 Celsius. Then add in, all at once, 18 milliliters of 99% tin-IV-chloride while rapidly stirring the indane/methylene chloride mixture. Note: during the addition of the tin-IV-chloride, maintain the temperature of the indan/methylene chloride mixture under 5 Celsius. After the addition of the tin-IV-chloride, immediately add in, in small portions at a time, 12 grams of dichloromethyl methyl ether over a period of about 15 minutes. During the addition, rapidly stir the reaction mixture, and maintain its temperature below 5 Celsius at all times. After the addition of the tin-IV-chloride, continue to rapidly stir the reaction mixture for an additional 30 minutes, and thereafter, remove the ice bath, and allow it to warm to room temperature. While the reaction mixture is warming to room temperature, add in 150 milliliters of ice water, and then moderately stir the reaction mixture as it warms up. Thereafter, place the entire reaction mixture into a seperatory funnel, and drain-off the lower methylene chloride layer. Note: the upper aqueous layer can be recycled or discarded if desired. After collecting the lower methylene chloride layer, wash this lower methylene chloride layer with three 100-millliter portions of ice cold water, followed by three 50-milliliter portions of a 5% hydrochloric acid solution, and then with two 50-milliliter portions of a 23% sodium chloride solution. After all the washings, the methylene chloride will be the lower layer each time. Now, place this methylene chloride portion into a suitable sized beaker, and then add in 10 grams of powdered charcoal, and then stir the entire mixture for about 10 minutes—thereafter, filter-off the charcoal. Note: use a filter paper that has a thin layer of celite, followed by a thin layer of silica gel placed there upon to better help remove the charcoal. After removing the charcoal through filtration, dry this washed methylene chloride portion by adding to it, 15 grams of anhydrous magnesium sulfate, and then stir the entire mixture for about 10 minutes—thereafter, filter-off the magnesium sulfate. Then place this filtered methylene chloride portion into a distillation apparatus, and distill-off the methylene chloride at 40 Celsius. When no more methylene chloride passes over or is collected, stop the distillation process, and recover the left over oily residue (after it has cooled), and then set it aside for step 2. The result will be about 14 grams of the desired product as a oily yellow liquid.

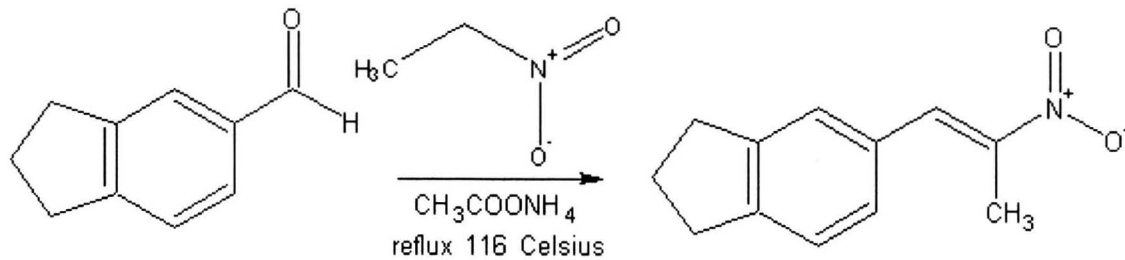


Indan

Indan-5-aldehyde

Step 2: Preparation of the nitro intermediate

Into a suitable sized reflux apparatus, equipped with motorized stirrer or other stirring means, place 12 grams of the product obtained in step 1, followed by 90 milliliters of nitroethane, and then followed by 3.6 grams of anhydrous ammonium acetate. Thereafter, reflux this entire mixture for about 8 hours at 116 Celsius with moderate stirring. Now, after refluxing the reaction mixture for about 8 hours, remove the heat source, and allow the reaction mixture to cool to room temperature. Thereafter, place this reaction mixture into an ice bath (or freezer), and chill to about 0 Celsius. Then let the reaction mixture stand at 0 Celsius for about 1 hour, and then filter-off any precipitated crystals of the nitro intermediate—then vacuum dry or air-dry these crystals, and then save them for later. Note: in some cases, some or very few crystals of the nitro intermediate may or may not precipitate when the reaction mixture is placed into the ice bath. In any case, place the remaining filtered reaction mixture (after letting it stand at 0 Celsius), into a distillation apparatus, and then carefully and slowly distil-off the excess nitroethane at 116 Celsius—water may be added to provoke volatization of the nitroethane through steam distillation; in this case, simply add in 75 to 100 milliliters of water and distill at 100 to 115 Celsius. Note: distillation under vacuum works best, and distillation at 116 Celsius may result in some or partial decomposition of the nitro product. When no more nitroethane passes over or is collected, stop the distillation process, and recover the left over remaining oily residue (after it has cooled). Then, add to this recovered left over oily residue, any dried filtered-off crystals (obtained earlier), and then extract this entire combined oily residue with three 50-millilter portions of methylene chloride, and after the extraction process, combine all methylene chloride portions, if not already done so, and then dry this combined methylene chloride portion, by adding to it, 10 grams of anhydrous magnesium sulfate. Note: after each extraction, the methylene chloride will be the lower layer each time, but the methylene chloride in most cases can simply be decanted-off rather then separated using a seperatory funnel. After adding in the anhydrous magnesium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Finally, place this filtered methylene chloride portion into a distillation apparatus, and distill-off the methylene chloride at 40 Celsius. When no more methylene chloride passes over or is collected, stop the distillation process, and then recover the left over remaining oily residue (after it has cooled). Then recrystallize this oily residue from 100 milliliters of methyl alcohol. Note: during the recrystallization process, the methyl alcohol, after boiling and concentration, needs to be placed into an ice bath (or freezer) and chilled to 0 Celsius for at least an hour or more (after it has cooled to room temperature), to allow the crystals of the nitro intermediate to properly crystallize, as they have a melting point of 34 Celsius and are difficult to crystallize from methyl alcohol. After the recrystallization process, and resulting codlings, vacuum dry or air-dry the filtered-off crystals of the nitro intermediate, and then place these crystals into an amber glass bottle, and store in a refrigerator until use.



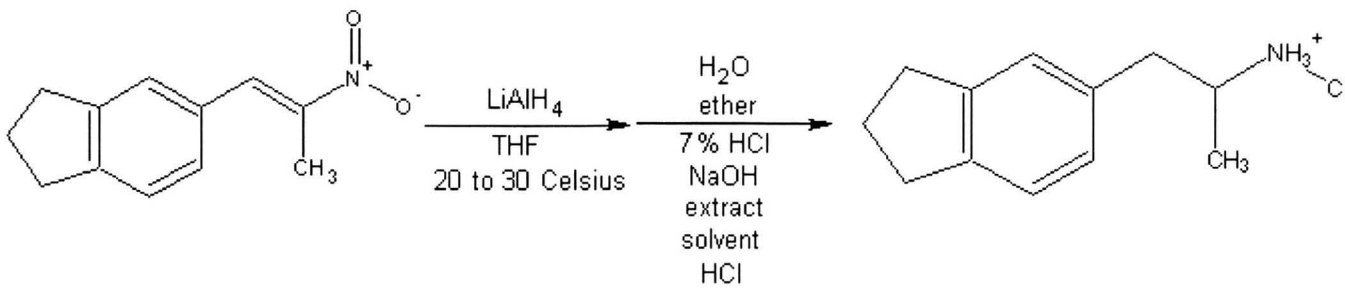
Indan-5-aldehyde

Indanyl nitro intermediate

Step 3: Preparation of Indanylamphetamine hydrochloride

Into a regular flask or beaker, place 11.6 grams of the product obtained in step 2, followed by 400 milliliters of dry tetrahydrofuran. Then stir the entire mixture briefly to form a uniform mix. Then, into a separate flask or beaker, equipped with motorized stirrer or other stirring means, place 6 grams of lithium aluminum hydride, followed by 600 milliliters of dry tetrahydrofuran. Then briefly stir this entire mixture to form a uniform mix. Then, slowly add, in small portions at a time, the tetrahydrofuran mixture of the product obtained in step 2, over a period sufficient to keep the lithium aluminum hydride mixture at around 20 to 30 celsius at all times. During the addition, rapidly stir the lithium aluminum hydride mixture. After the addition, continue to stir the reaction mixture at room temperature for about 10 hours. After 10 hours, slowly and carefully add in, 40 milliliters of ice-cold water (to destroy any lithium salts), and then stir the entire diluted reaction mixture for about 30 minutes. Then filter-off any insoluble solids, and then briefly wash the filtered-off solids with two 25-millilter portions of diethyl ether, and after the washings, combine the ether washing portions with the filtered reaction mixture. Now, place this reaction mixture into a distillation apparatus, and distill-off the water, tetrahydrofuran, and ether by distillation at 100 Celsius. When no more water, tetrahydrofuran, or ether passes over or is collected, stop the distillation process, and recover the let over remaining residue (after it has cooled). Then, mix this collected leftover residue with 400 milliliters of diethyl ether, and then stir the mixture for about 10 minutes. Then, extract this ether mixture with ten 50-milliliter portions of a 7% hydrochloric acid solution, and after the extraction process, combine all acidic aqueous portions (if not already done so). Note: after each

extraction, the acidic aqueous portion will be the lower layer each time—after the extraction process, the upper ether layer can be recycled or discarded if desired. Now, place this combined acidic aqueous portion into a clean suitable sized beaker, and then add in, a sodium hydroxide solution prepared by adding and dissolving 45 grams of sodium hydroxide into 150 milliliters of cold water. Note: sodium hydroxide generates excessive heat when dissolved in water, so allow the alkaline solution to cool to room temperature before using. After adding in the sodium hydroxide solution, stir the entire mixture for about 30 minutes. After 30 minutes, extract the entire alkaline mixture with six 50-milliliter portions of methylene chloride, and after the extraction process, combine all methylene chloride portions, if not already done so, and then dry this combined methylene chloride portion by adding to it, 15 grams of anhydrous magnesium sulfate. Note: after the extraction process, the methylene chloride will be the lower layer each time. After adding in the anhydrous magnesium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Finally, place this dried filtered methylene chloride portion into a distillation apparatus, and distil-off the methylene chloride at 40 Celsius. When no more methylene chloride passes over or is collected, stop the distillation process, and recover the left over remaining residue (after it has cooled). Then dissolve this recovered left over residue into 80 milliliters of diethyl ether, and then stir the entire mixture for about 10 minutes—then quickly filter the mixture to remove any potential insoluble materials (if any). Then place this filtered ether mixture into an ice bath, and chill to about 0 Celsius. Thereafter, bubble into this ether mixture, 15 grams of hydrogen chloride gas (excess). After the addition of the hydrogen chloride, allow the mixture to stand for about 1 hour at 0 Celsius, then filter-off the precipitated crystals of the desired Indanylamphetamine, and then vacuum dry or air-dry the crystals. The result will be about 8 grams of the desired product.

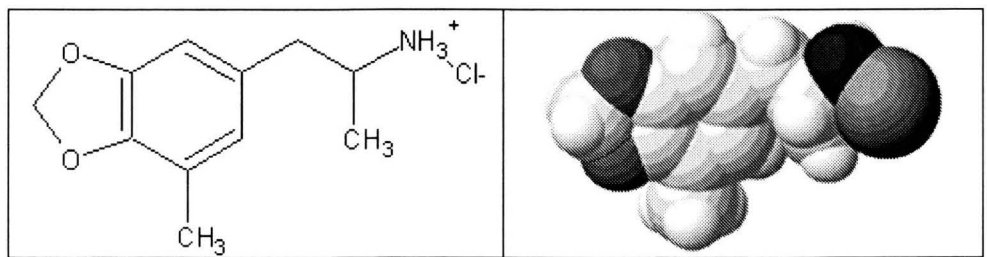


Indanyl nitro intermediate

IAP

Note: other salts of the freebase IAP can be prepared in the usual manner, by replacing the hydrogen chloride gas with 98% sulfuric acid, pure tartaric acid, citric acid, or 80% phosphoric acid.

0035. Methyl MDA. 5-methyl-MDA. *1-(7-methyl-1,3-benzodioxol-5-yl)propan-2-amine*



Structure | 3D model

Methyl MDA forms colorless to white crystals with a melting point of 215 Celsius. The crystals are soluble in water and alcohol, but insoluble in the usual organic solvents. Methyl MDA is very similar to MDA in its structural and physical integrity, but its effects are much different. It has the ability to mimic effects produced by LSD and MDMA, making it a combination of a psychedelic amphetamine and a classic hallucinogen. Human trials of this drug are rare, and its use by humans is very limited, probably due to its pain staking preparation. However, a few human trials with methyl MDA has demonstrated and shown the drug to produce an outstanding array of effects ranging from the usual enhancements of sight, sound, touch, and feelings, leading up to vivid out-of-body like trips, to strange and mythological effects ranging from emotional enhancements, feelings of euphoria, well-being, and strange sight and vision effects similar to straight LSD consumption—many of these effects can be hard to describe, and may very in individual users. Regardless of the expense in manufacture of this drug, methyl MDA does show unprecedented potential as a remarkable psychedelic amphetamine for street consumption—that has an added LSD like kick. Note: This drug has been highly recommended for experimental use in animal and human trials by retired or late scientists.

This substance is a controlled substance (psychedelic/hallucinogenic amphetamine) as listed in the US code of Federal regulations.

Toxicity: Low	**Rate of onset (average):** Unknown
Stimulation dosage (ingestion): 15 to 30 milligrams	**Duration of effects (average):** Unknown—may last up to 12 hours
Stimulation dosage (inhalation): unknown	**Habit forming potential:** Very low
Stimulation dosage (injection): 3 to 6 milligrams	**Estimated value U.S. (based on procedure):** $29 to $35 per gram. Street value could be $5.00 per 20 milligram hit, which would equal $250 per gram

Procedure A: Preparation of Methyl MDA

Materials:

1. 12 grams of a 37% formaldehyde solution	16. 15 grams of pyridine
2. 18 grams of a 40% dimethylamine solution	17. 150 milliliters of a 15% hydrochloric acid solution
3. 90 milliliters of 95% ethyl alcohol	18. 50 milliliters of cyclohexane
4. 15 grams of vanillin (see intermediate-0019. 3,4,5-TMB, procedure B, step 1)	19. 325 milliliters of ethyl acetate
5. 50 milliliters of acetone	20. 16.5 grams of cesium carbonate
6. 62 milliliters of acetic anhydride	21. 90 milliliters of dimethylformamide (DMF)
7. 90 milliliters of 35 to 38% hydrochloric acid	22. 6.5 grams of bromochloromethane
8. 50 milliliters of para-dioxane	23. 16 milliliters of nitroethane
9. 42 grams of tin-II-chloride dihydrate	24. 100 milliliters of methyl alcohol
10. 295 milliliters of methylene chloride	25. 16 milliliters of toluene
11. 300 milliliters of a 10% hydrochloric acid solution	26. 5.3 grams of lithium aluminum hydride
12. 125 milliliters of a 23% sodium chloride solution	27. 90 milliliters of tetrahydrofuran
12. 60 grams of anhydrous magnesium sulfate	28. 24 milliliters of 99% isopropyl alcohol
13. 25 grams of silica gel of 60 to 210 mesh	29. 4 milliliters of a 15% sodium hydroxide solution
14. 600 milliliters of diethyl ether	30. 30 grams of sodium hydroxide
15. 100 milliliters of hexane	31. 10 grams of hydrogen chloride gas

Summary: Methyl MDA is prepared in an exhaustive six-step process starting with the formation of 5-dimethylaminomethyl-4-hydroxy-3-methoxybenzaldehyde. This intermediate is readily prepared by reacting vanillin with formaldehyde and dimethylamine in the presence of 95% ethyl alcohol. The reaction is very mild, and thereafter, it is stirred for a prolonged period of time. The next day, the precipitated crystals are then filtered-off, washed, and then dried. The resulting product of 5-dimethylaminomethyl-4-hydroxy-3-methoxybenzaldehyde is then converted into 4-hydroxy-3-methoxy-5-methylbenzaldehyde by reaction with acetic anhydride under reflux conditions. After the reaction, the acetylated pre-intermediate is then converted into the desired product of 4-hydroxy-3-methoxy-5-methylbenzaldehyde by refluxing with tin-II-chloride catalyst, in the presence of a suitable solvent. After this reaction, the desired product is thus obtained by an exhaustive purification process utilizing solvent extraction, and then treatment with silica gel in the usual manner. After the exhaustive purification and work-up process, the desired product is then collected by evaporation of the solvent. The final desired product can then be sublimed in the usual manner to obtain a purified product of 4-hydroxy-3-methoxy-5-methylbenzaldehyde. This 4-hydroxy-3-methoxy-5-methylbenzaldehyde is then converted into 3,4-dihydroxy-5-methylbenzaldehyde by reaction with anhydrous aluminum chloride in the presence of pyridine and methylene chloride. After heating the reaction mixture to reflux, and cooling, the resulting mixture is treated with acid (to hydrolyses any product), and the resulting mixture is then separated (to recover the upper acidic aqueous layer), which is then extracted into ether. The ether is then removed in the usual manner, and the left over residue is then collected. The collected desired product of 3,4-dihydroxy-5-methylbenzaldehyde is then converted into 5-methyl-piperonal by reaction with cesium carbonate and bromochloromethane in the presence of dimethylformamide (DMF). After the reaction, the desired product is obtained by evaporation of the reaction mixture, followed by dilution of the recovered residue into water, followed by extraction with solvent. The solvent is removed under the usual means, and the left over residue is then recovered. This left over residue is the desired 5-methyl-piperonal, which is then converted into 3,4-methylenedioxy-5-methylphenyl-2-nitropropene by condensation with nitroethane in the usual manner. The reaction is refluxed and afterwards, evaporated, and the left over residue then dissoluted into ether, and this ether mixture is then removed, and the left over residue is then recrystallized from methyl alcohol. The resulting 3,4-methylenedioxy-5-methylphenyl-2-nitropropene is then finally converted into the desired methyl MDA by reduction with lithium aluminum hydride in a familiar manner. After the reduction, the reaction mixture is treated with the usual reagents (to remove lithium salts), and the resulting reaction mixture is then evaporated. The left over residue is then treated with acid, washed with ether, and then treated with base. The basified mixture is then extracted into suitable solvent, and the resulting mixture is then evaporated. The left over residue is then dissolved into alcohol, and the desired methyl MDA is then precipitated by the addition of hydrogen chloride in the usual manner.

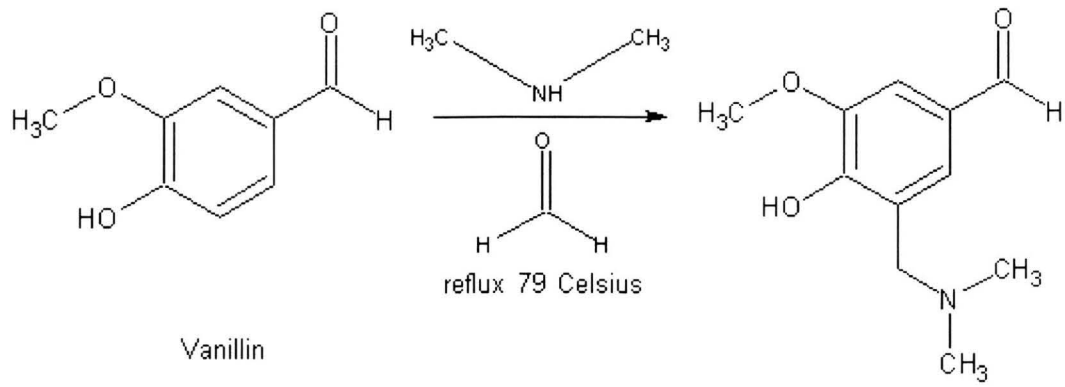
Hazards: Extinguish all flames before using diethyl ether, tetrahydrofuran, nitroethane, acetone, dioxane, hexane, cyclohexane, and ethyl acetate, all of which are highly flammable and most can form explosive mixtures with air. Wear gloves and use caution when handling lithium aluminum hydride, and avoid contact with water. Wear gloves when handling sodium hydroxide, hydrochloric acid, hydrogen chloride gas, and acetic anhydride, as they can cause skin irritation—hydrogen chloride gas is an irritating gas so avoid inhalation. Wear gloves and use proper ventilation when handling concentrated formaldehyde solution, and concentrated dimethylamine solution—avoid inhalation and skin contact. Use proper ventilation when handling toluene, as it is a suspected carcinogen. Methyl alcohol burns with a colorless flame, so extinguish all sources of ignition before using—as burning methanol cannot be seen, and hence, gives no warning of its presence. Cesium carbonate is toxic, so wear gloves when handling and avoid inhalation of the dust.

Procedure:

Personnel notes for procedure A: Methyl MDA

Step 1: Preparation of 5-dimethylaminomethyl-4-hydroxy-3-methoxybenzaldehyde

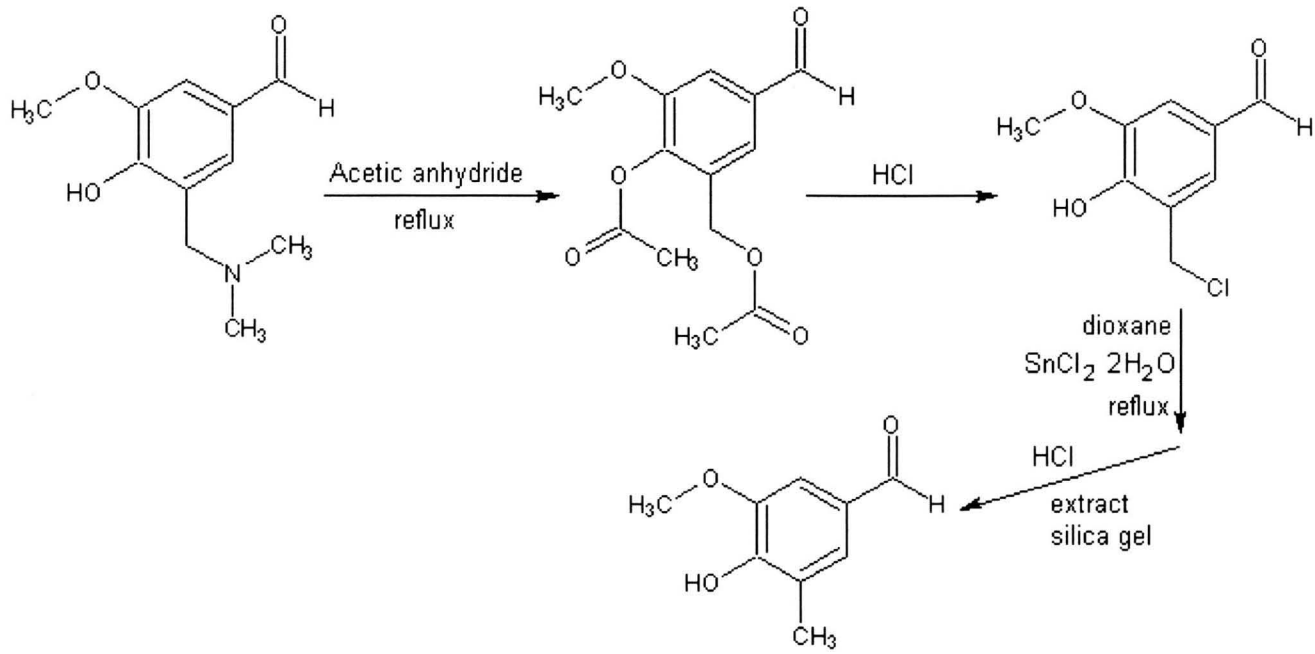
Into a suitable sized reflux apparatus (equipped with motorized stirrer or other stirring means, and thermometer), place 12 grams of a 37% formaldehyde solution, followed by 18 grams of a 40% dimethylamine solution, and then followed by 90 milliliters of 95% ethyl alcohol. Then stir the entire mixture for about 10 minutes to form a uniform mix. Thereafter, add in, in small portions at a time, 15 grams of vanillin (see intermediate-0019. 3,4,5-TMB, procedure B, step 1), and then rapidly blend the mixture for about 10 minutes. Then reflux the entire mixture at 79 Celsius for about 30 minutes. After 30 minutes, remove the heat source, and allow the reaction mixture to cool to about 25 Celsius, and then stir the reaction for 24 hours at room temperature. Thereafter, place the reaction mixture into a refrigerator, and then allow it to stand overnight. The next day, remove the reaction mixture from the refrigerator, and filter-off the white granular precipitate Then wash this filtered-off granular precipitate with two 25-millilter portions of ice cold acetone (several times using the same washing portion), and then vacuum dry or air-dry the washed filtered-off precipitate. The result will be about 17 grams of the desired product with a melting point of 141 Celsius.



Step 2: Preparation of 4-hydroxy-3-methoxy-5-methylbenzaldehyde

Into a standard reflux apparatus, place 13 grams of the product obtained in step 1, followed by 62 milliliters of acetic anhydride, and then reflux the entire mixture at 139 Celsius for about 3 hours. Note: place a calcium chloride drying tube over the reflux condenser to exclude moisture. After 3 hours, remove the heat source, and allow the refluxed reaction mixture to cool to room temperature. Thereafter, pour the entire reaction mixture into a shallow pan, with a high surface area, and allow the reaction mixture to evaporate. Note: blowing air over the surface of the reaction mixture using a conventional cooling fan, can help speed up the evaporation process. Thereafter, when all the liquid has been removed, and only a residue remains, recover the left over remaining residue, and then place this recovered residue into a suitable sized beaker, and then add in, 65 milliliters of 35 to 38% hydrochloric acid (muriatic acid will work). Then rapidly stir the entire acidic mixture for about 1 hour at room temperature. After rapidly stirring the acidic mixture for about 1 hour, filter-off any precipitated solids, and then vacuum dry or air-dry these filtered-off solids. Now, place these dried filtered-off solids into a suitable sized beaker, and then add in 50 milliliters of para-dioxane, and then place the entire mixture into a reflux apparatus (equipped with motorized stirrer),

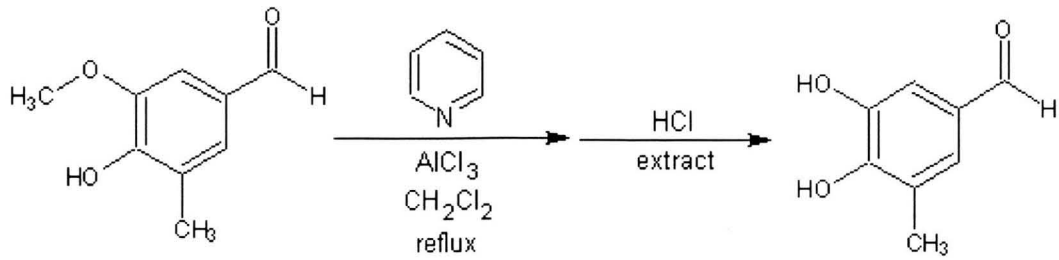
and gently heat to about 60 Celsius. Then, add in, 42 grams of tin-II-chloride dihydrate, in small portions at a time, over a period of about 5 minutes (add through the top of the reflux condenser). During the addition of the tin-II-chloride dihydrate, rapidly stir the reaction mixture. After the addition of the tin-II-chloride dihydrate, reflux the entire reaction mixture at 101 Celsius for about 30 minutes. After the refluxing period, remove the heat source, and allow the reaction mixture to cool to room temperature. Thereafter, pour the entire reaction mixture into a suitable sized beaker, and then add in 25 milliliters of 35 to 38% hydrochloric acid (muriatic acid will work), and then stir the entire mixture for about 30 minutes. Then extract this entire acidic reaction mixture with five 25-milliliter portions of methylene chloride, and after the exaction process, combine all methylene chloride portions (if not already done so), and then wash this combined methylene chloride portion with three 50-milliliter portions of a 10% hydrochloric acid solution, followed by three 50-milliters portions of ice cold water, and then followed by three 25-milliliter portions of a 23% sodium chloride solution. Note: after the extraction and washings, the methylene chloride will be the lower layer each time. After washing the combined methylene chloride portion, dry this methylene chloride portion by adding to it, 15 grams of anhydrous magnesium sulfate, and then stir the entire mixture for about 10 minutes—thereafter, filter-off the magnesium sulfate. Then place this filtered methylene chloride portion into a distillation apparatus, and distill-off the methylene chloride at 40 Celsius. When no more methylene chloride passes over or is collected, stop the distillation process, and recover the left over remaining residue (after it has cooled). Now, dissolve this residue into 100 milliliters of fresh methylene chloride, and then pass this methylene chloride mixture through a glass column filled with 10 grams of silica gel of 60 to 210 mesh (a standard silica gel column), several times. Afterwards, place this methylene chloride mixture (which was passed through the silica gel column several times), into a distillation apparatus, and distill-off the methylene chloride at 40 Celsius. Again, when no more methylene chloride passes over or is collected, stop the distillation, and recover the left over remaining residue (after it has cooled). Then dissolve this left over residue into a solvent mixture prepared by adding 10 milliliters of methylene chloride to 50 milliliters of diethyl ether and then adding in 100 milliliters of hexane. Then pass this solvent mixture through a clean glass column filled with 15 grams of silica gel of 60 to 210 mesh (another standard silica gel column), several times. Afterwards, place this solvent mixture (which was passed through the silica gel column several times), into a distillation apparatus, and distill-off the methylene chloride, hexane, and ether by distillation at 69 Celsius. When no more methylene chloride, hexane, or ether passes over or is collected, stop the distillation process, and recover the left over remaining residue (after it has cooled). Finally, place this left over remaining residue aside for step 3. Note: this gray recovered residue can be sublimed at 120 Celsius, using the normal sublimation techniques, to obtain a purified desired product as a white solid with a melting point of 101 Celsius.



Step 3: Preparation of 3,4-dihydroxy-5-methylbenzaldehyde

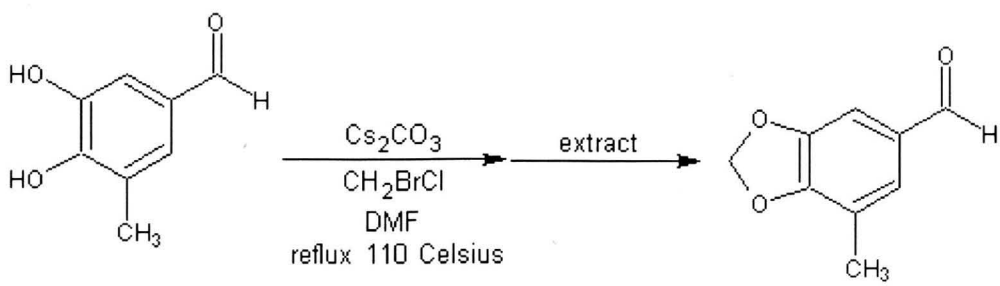
Into a suitable reflux apparatus, equipped with motorized stirrer or other stirring means and addition funnel, place 6.4 grams of anhydrous aluminum chloride, followed by 7 grams of the product obtained in step 2, and then followed by 60 milliliters of methylene chloride. Then place a calcium chloride drying tube over the reflux condenser to keep moisture out. Then stir the entire mixture for about 10 minutes to form a uniform mix, and then place 15 grams of pyridine into to the addition funnel. Thereafter, gently heat the reaction mixture to about 35 celsius, and when its temperature reaches 35 celsius, add in the pyridine contained in the addition funnel, drop-wise, over a period sufficient to keep the reaction mixture below 40 Celsius at all times. During the addition, rapidly stir the reaction mixture. After the addition of the pyridine, continue to stir the reaction

mixture for about 10 minutes, and then reflux the entire reaction mixture at about 40 to 50 Celsius for about 6 hours with moderate stirring. After refluxing for about 6 hours, remove the heat source, and allow the reaction mixture to cool to room temperature. Then pour the entire reaction mixture into a suitable sized beaker, and then slowly add in, drop-wise, 150 milliliters of a 15% hydrochloric acid solution. During the addition of the acid, rapidly stir the reaction mixture. After the addition of the hydrochloric acid, place the entire two-phase reaction mixture into a seperatory funnel, and remove the upper acidic aqueous layer. Then extract this entire collected acidic aqueous layer with three 50-millilter portions of diethyl ether, and after the extraction process, combine all ether portions (if not already done so), and then dry this combined ether portion, by adding to it, 10 grams of anhydrous magnesium sulfate. Note: after each extraction, the ether will be the upper layer each time. After adding in the magnesium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Finally, place this filtered ether mixture into a distillation apparatus, and distill-off the ether at 40 Celsius. When no more ether passes over or is collected, stop the distillation process, and recover the left over remaining residue (after it has cooled). Then recrystallize this collected left over residue from a solvent mixture prepared by adding and dissolving 50 milliliters of ethyl acetate into 50 milliliters of cyclohexane. After the recrystallization process, vacuum dry or air-dry the filtered-off crystals.



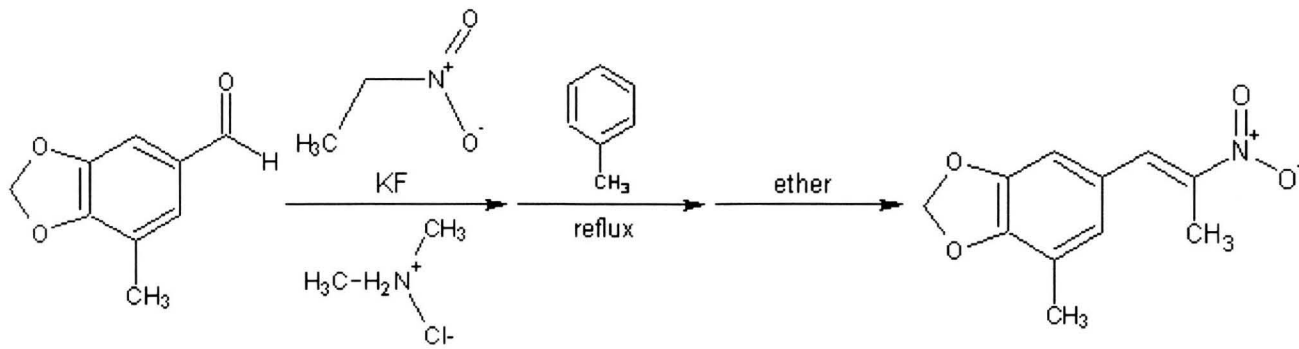
Step 4: Preparation of 5-methyl-piperonal

Into a clean reflux apparatus, equipped with motorized stirrer or other stirring means, place 5 grams of the product obtained in step 3, followed by 16.5 grams of cesium carbonate, and then followed by 90 milliliters of dimethylformamide. Thereafter, stir the entire mixture for about 10 minutes to form a uniform mix. Then add in, 6.5 grams of bromochloromethane, and then reflux the entire mixture at 110 Celsius for about 90 minutes. After 90 minutes, stop the reflux process, and allow the reaction mixture to cool to room temperature. Afterwards, filter the reaction mixture to remove any precipitated impurities, and then briefly wash these filtered-off solids with two 25-millilter portions of ethyl acetate. After the washing, combine both ethyl acetate washing portions to the filtered reaction mixture. Note: the reaction mixture should be filtered by placing a thin layer a celite over the filter paper prior to filtering the reaction mixture to aid in separation of the precipitated impurities. Now, place the combined reaction mixture into a shallow pan, and allow it to evaporate. Note: blowing air over the surface of the shallow pan using a conventional cooling fan can be used to speed up the evaporation process. Second note: using a vacuum distillation apparatus works best and is effective at recovering the dimethylformamide solvent. After the evaporation process, collect the left over remaining residue, and then place it into a clean beaker. Thereafter, add in, 100 milliliters of warm water and then stir the entire mixture for about 30 minutes. Then extract this entire mixture with three 75-milliliter portions of ethyl acetate, and after the extraction process, combine all ethyl acetate portions, if not already done so, and then wash this combined ethyl acetate portion with two 25-milliliter portions of cold water, followed by two 25-milliliter portions of a 23% sodium chloride solution. Note: after each extraction and washing portion, the ethyl acetate will be the upper layer each time. Now, dry this washed combined ethyl acetate portion by adding to it, 15 grams of anhydrous magnesium sulfate, and then stir the entire mixture for about 10 minutes—thereafter, filter-off the magnesium sulfate. Finally, place this filtered ethyl acetate mixture into a distillation apparatus, and distill-off the ethyl acetate at 77 Celsius. When no more ethyl acetate passes over or is collected, stop the heating process, and then recover the left over remaining residue (after it has cooled). Then set aside this left over residue for step 5.



Step 5: Preparation of 3,4-methylenedioxy-5-methylphenyl-2-nitropropene

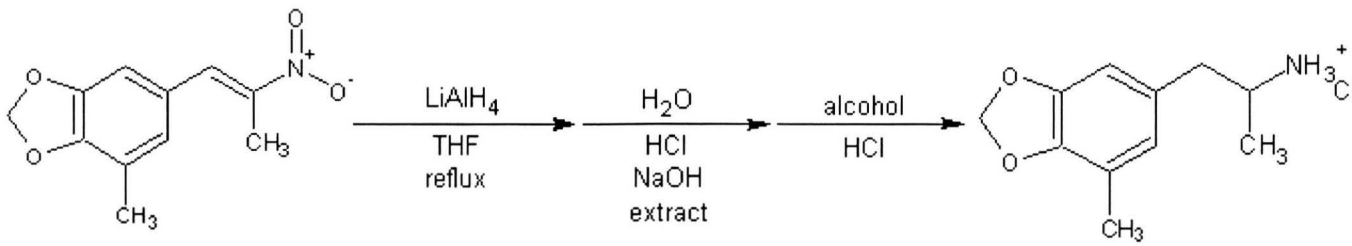
Into a standard reflux apparatus, equipped with motorized stirrer or other stirring means, place 4.4 grams of the product obtained in step 4, followed by 16 milliliters of nitroethane, then followed by 4 grams of dimethylamine hydrochloride, then followed by 230 milligrams of potassium fluoride, and then finally followed by 16 milliliters of toluene. Note: place a calcium chloride drying tube over the reflux condenser to exclude moisture. Thereafter, reflux this entire mixture at 116 Celsius for about 12 hours with rapid stirring. After refluxing the reaction mixture for 12 hours, quickly remove the reflux condenser, and replace it with a conventional cold-water condenser (fitted with receiver flask), and then distill-off all liquids at 116 Celsius. When no more liquids pass over or are collected, stop the distillation process, and recover the left over remaining residue (after it has cooled). Then place this collected left over residue into a clean beaker, and then add in 75 milliliters of cold water, followed by 75 milliliters of diethyl ether. Thereafter, stir the entire mixture for about 30 minutes, and then place the entire two-phase mixture into a seperatory funnel, and then allow it to stand for about 10 minutes—then remove the upper ether layer; after removing the lower water layer first. Then dry this colleted ether layer, by adding to it, 10 grams of anhydrous magnesium sulfate, and then stir the entire mixture for about 10 minutes—thereafter, filter-off the magnesium sulfate. Finally, place this dried filtered ether layer into a distillation apparatus, and remove the ether by distillation at 40 Celsius. When no more ether passes over or is collected, stop the distillation process, and recover the left over remaining residue (after it has cooled). Then recrystallize this dried collected residue from 100 milliliters of methyl alcohol, and after the recrystallization process, vacuum dry or air-dry the collected crystals. The result will be about 5 grams of the desired product as pale yellow crystals.



Step 6: Preparation of methyl MDA

Into a reflux apparatus, equipped with motorized stirrer or other stirring means, and addition funnel, place 5.3 grams of lithium aluminum hydride, followed by 50 milliliters of tetrahydrofuran. Then stir the entire mixture to form a uniform mixture. Thereafter, prepare a solution by adding and dissolving 4.4 grams of the product obtained in step 5, into 40 milliliters of tetrahydrofuran. Then place this solution into the addition funnel, and then add this solution, drop-wise, to the lithium aluminum hydride mixture over a period of about 5 to 10 minutes. After the addition, reflux the entire reaction mixture at 68 Celsius for 10 hours while rapidly stirring the reaction mixture. After the reflux period, remove the heat source, and allow the reaction mixture to cool to room temperature. Then place this reaction mixture into a suitable sized beaker, and then slowly and carefully add in, 4 milliliters of 99% isopropyl alcohol, followed carefully by 4 milliliters of a 15% sodium hydroxide solution, and then followed carefully by 14 milliliters of ice cold water. During each addition, rapidly stir the reaction mixture. After all additions, stir the entire reaction mixture for about 30 minutes, and then filter-off the precipitated impurities. Now, place this filtered reaction mixture into a distillation apparatus, and distill-off the tetrahydrofuran at 68 Celsius. When no more tetrahydrofuran passes over or is collected, stop the distillation process, and recover the left over remaining residue (after it has cooled). Then place the recovered collected residue into a suitable beaker, and then add in 100 milliliters of cold water, followed by 150 milliliters of a 10% hydrochloric acid solution, and then stir the entire mixture for about 1 hour. Then wash this acidic mixture with two 50-milliliter portions of diethyl ether. After each washing, the acidic mixture will be the lower layer each time—the upper ether layers can be recycled if desired. After the washing, place the washed lower acidic portion into a clean beaker, and then add in a sodium hydroxide solution prepared by adding and dissolving 30 grams of sodium hydroxide into 150 milliliters of cold water. Note: sodium hydroxide generates excessive heat when dissolved in water, so allow the alkaline solution to cool before using. After adding in the sodium hydroxide solution, rapidly blend the entire now alkaline mixture for about 1 hour. Finally, extract this alkaline mixture with three 50-milliliter portions of diethyl ether, and after the extraction process, combine all ether layers, if not already done so, and then dry this combined ether layer by adding to it, 10 grams of anhydrous magnesium sulfate. Note: after each extraction, the ether will be the upper layer each time. After adding in the magnesium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Then place this filtered ether mixture into a distillation apparatus, and distill-off the ether at 40 Celsius. When no more ether passes over or is collected, stop the distillation process, and collect the left over remaining residue (after it has cooled). Now, dissolve this recovered residue into 20 milliliters of 99% isopropyl alcohol, and then stir the entire mixture for about 10 minutes—then briefly filter-off any insoluble impurities, and then place this filtered alcohol mixture into an ice bath, and then bubble into it, 10 grams of hydrogen chloride gas. After the addition of the hydrogen chloride gas, add in 25 milliliters of diethyl ether, and then allow the entire mixture to stand at 0 Celsius for about 1 hour. Thereafter, filter-off the precipitated crystals of the desired

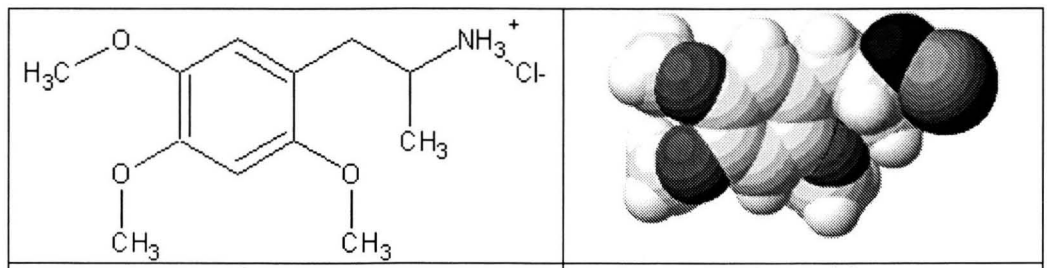
methyl MDA, then wash these crystals with two 25-milliter portions of diethyl ether, and then vacuum dry or air-dry the crystals of the methyl MDA.



methyl MDA

Note: other salts of the freebase methyl MDA can be prepared in the usual manner, by replacing the hydrogen chloride gas with 98% sulfuric acid, pure tartaric acid, citric acid, or 80% phosphoric acid.

0036. TMA2. 2,4,5-Trimethoxyamphetamine hydrochloride. *1-(2,4,5-trimethoxyphenyl)propan-2-amine hydrochloride*



Structure | 3D model

TMA2 forms colorless to white crystals with a melting point of 189 Celsius. The crystals are soluble in water and alcohol, but insoluble in the usual organic solvents. TMA2 is a typical psychedelic amphetamine with the usual body effects. The major difference between TMA2 and other psychedelic amphetamines is the potency. TMA2 is quite potent and much more powerful then MESCALINE or similar compounds. Ingestion of TMA2 produces a variety of effects, one of which being strange and vivid motions to objects in the surrounding world—many of these motions can vary, but usually are fantastic and very pleasant. For example, certain objects like statues in the real world, will appear to have flaming and/or strange motion trails or added features such as rolling flesh, wavy feathers blowing in the wind, and overall shiny to wavy appearances. TMA2 increases the feelings to music, and music and other forms of musical entertainments are enhanced primarily on the psychological level. Secondary effects of TMA2 include, a hypnotic or relaxing state of mind, increases in mental awareness, enhancements to the usual sights, sounds, touches, and feelings, interesting enhancements to vision producing kaleidoscopic like imagery when the eyes are closed, and a happy or stupor-like attitude resulting in laughter and a abnormal sense of humor. In essence, TMA2 produces a very pleasant and peaceful trip, with only a few minor side effects. These side-effects may include slight nausea lasting for only the first couple of hours, mild cases of diarrhea, which usually subside after about the second hour, numbness in fingers and hands, and slight difficulty in sleeping—the later may exist in only 50% of users for the first couple of trips. Sleeping disorders usually disappear after several trips, as do most side effects.

This substance is a controlled substance (psychedelic amphetamine) as listed in the US code of Federal regulations.

Toxicity: Moderate	**Rate of onset (average):** Average—may take up to 1 hour for effects to be realized, but some cases have reported as long as 2 hours.
Stimulation dosage (ingestion): 20 to 40 milligrams	**Duration of effects (average):** 8 to 12 hours
Stimulation dosage (inhalation): 5 to 15 milligrams	**Habit forming potential:** Low
Stimulation dosage (injection): 5 to 9 milligrams	**Estimated value U.S. (based on procedure):** $21 to $25 per gram

Procedure A: Preparation of TMA2

Materials:

1. 1 kilogram of dried chopped up calamus root	10. 10 grams of iron fillings

2. 150 milliliters of a 20% sodium carbonate solution	11. 200 milligrams of anhydrous ferric chloride
3. 25 grams of anhydrous magnesium sulfate	12. 260 milliliters of toluene
4. 1005 milliliters of diethyl ether	13. 26 milliliters of 35 to 38% hydrochloric acid
5. 100 grams of sodium nitrite	14. 6 milliliters of formamide
6. 160 milliliters of a 25% sulfuric acid solution	15. 12 milliliters of 90% formic acid
7. 10 grams of anhydrous sodium carbonate	16. 20 grams of sodium hydroxide
8. 100 milliliters of 95% ethyl alcohol	17. 10 grams of dry hydrogen chloride gas
9. 200 milliliters of a 10% hydrochloric acid solution	

Summary: TMA2 can be prepared in a four-step process starting with the formation of 2,4,5-trimethoxy-2-nitropropene. This nitro intermediate is prepared from asarone by reaction with sodium nitrite in the presence of dilute sulfuric acid. The resulting nitro intermediate is then collected by first, allowing the reaction mixture to stand overnight, and the following day, the crystals of the nitro intermediate are then collected by simply filtering them off. The filtered-off crystals are then washed with water, and ether, and then dried. The dried nitro intermediate crystals are then dissolved into an alkaline alcohol solution, the resulting mixture is then acidified, and the resulting acidified mixture is then treated with ice, and allowed to stand. The precipitated refined crystals of the nitro intermediate are then filtered-off, and then vacuum dried. These purified crystals of the nitro intermediate are then converted into 2,4,5-trimethoxyphenyl-2-propanone by reduction with iron. Note: the asarone used to produce the nitro intermediate can be conveniently obtained by steam distillation of calamus root. Steam distillation of this root produces a yellowish oil, which can be purified by fractional distillation. The fractional distillation produces a refined asarone oil, which is composed of two primary isomers of asarone, also called asarones. This asarone product is then converted into the nitro intermediate by the just described process. The nitro intermediate (just previously mentioned) is then converted into 2,4,5-trimethoxyphenyl-2-propanone by reaction with iron, in the presence of toluene and hydrochloric acid. A small amount of ferric chloride is added to help initiate the reduction. After the chemical reaction is complete, the reaction mixture is drowned into water, and the resulting diluted mixture is then extracted with ether. The ether extracts are combined in the usual manner, and then evaporated to leave behind a residue. This residue is composed primarily of the desired 2,4,5-trimethoxyphenyl-2-propanone. This propanone intermediate is then finally converted into the desired TMA2 by reaction with formamide in the presence of a small amount of concentrated formic acid. The reaction mixture is heated and distilled for several hours, and the resulting distilled reaction mixture is then extracted with toluene. The combined toluene extracts are then evaporated in the usual manner, and the left over residue is then briefly refluxed with concentrated hydrochloric acid, and then treated with base. The basified mixture is then extracted with ether, and the combined ether extract is then gassed with hydrogen chloride in the usual manner to precipitate the desired TMA2 as the hydrochloride salt. The filtered ether mixture can then be evaporated to only 20% of its original volume to afford precipitation of additional crystals of TMA2.

Hazards: Use caution when handling diethyl ether, as it is capable of forming explosive mixtures with air—extinguish all flames before using. Wear gloves when handling sodium hydroxide, hydrochloric acid, sulfuric acid, and formic acid, as they are all capable of causing skin irritation—formic acid is capable of producing highly irritating skin burns. Use proper ventilation when handling toluene, which is a suspected carcinogen.

Preparation:

Personnel notes for procedure A: TMA2

Step 1: Isolation of Asarone from calamus root

Into a standard steam distillation apparatus (into the 3-neck distillation flask), place 1 kilogram of dried chopped up calamus root, followed by 1500 milliliters of water, and then steam distill the entire mixture at 100 Celsius for several hours until no more viscous yellowish oil is collected in the receiver flask. Note: the steam distillation process can take any where from 4 to 8 hours. After the steam distillation process, recover the contents in the receiver flask, and then place these contents into a suitable sized seperatory funnel, and then allow this seperatory funnel to stand for about 1 hour. Then recover the upper yellow liquid, after removing the lower water layer first. Note: in some cases the yellow oil may be the lower layer. Then wash this recovered yellow oil with three 50-milliliter portions of a 20% sodium carbonate solution, followed by three 50-millilter portions of warm water (the water should be heated to about 35 to 40 Celsius). After the washings, the yellow oil will be the upper layer each time. Note: in some cases, the yellow oil may be the lower layer. After the washings, dry this yellow oil by

adding to it, 5 grams of anhydrous magnesium sulfate, and then stir the entire mixture for about 10 minutes—thereafter, filter-off the magnesium sulfate.

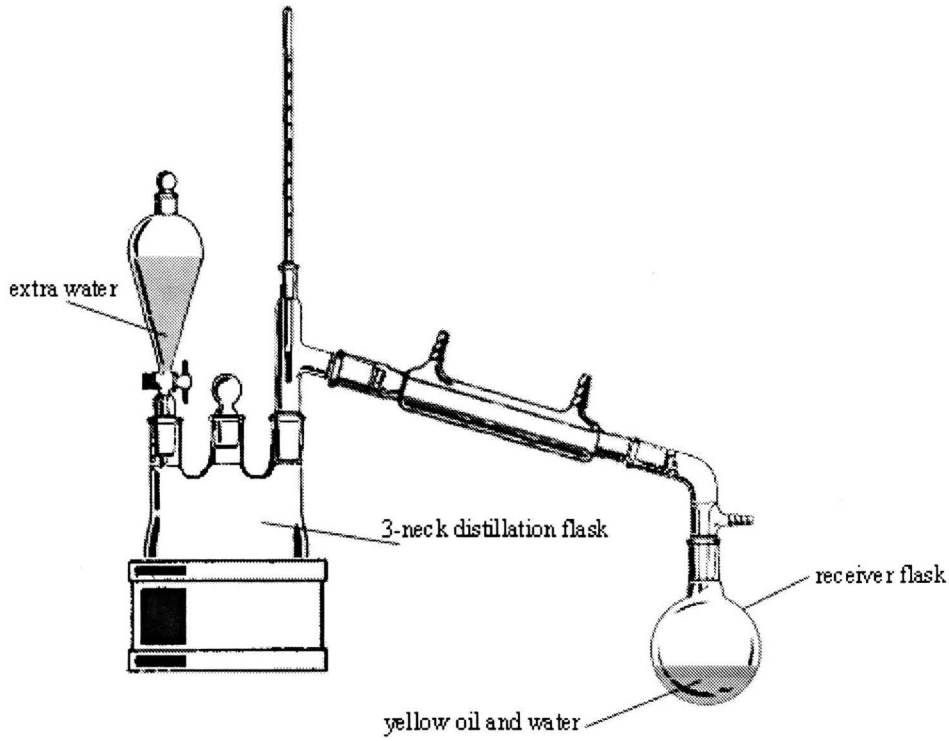


Figure 049. Standard steam distillation apparatus with steam generated internally by heating to 100 Celsius.

After the steam distillation process, and after the recovered yellow oil has been washed, it needs to be purified by fractional distillation. Setup-up the distillation apparatus as in the following illustration, and then fractionally distill the yellow oil at 200 Celsius, until no more oil passes over or is collected in the receiver flask. After the fractional distillation process, the result will be about 21 grams of the purified asarone, as a colorless to yellowish oil.

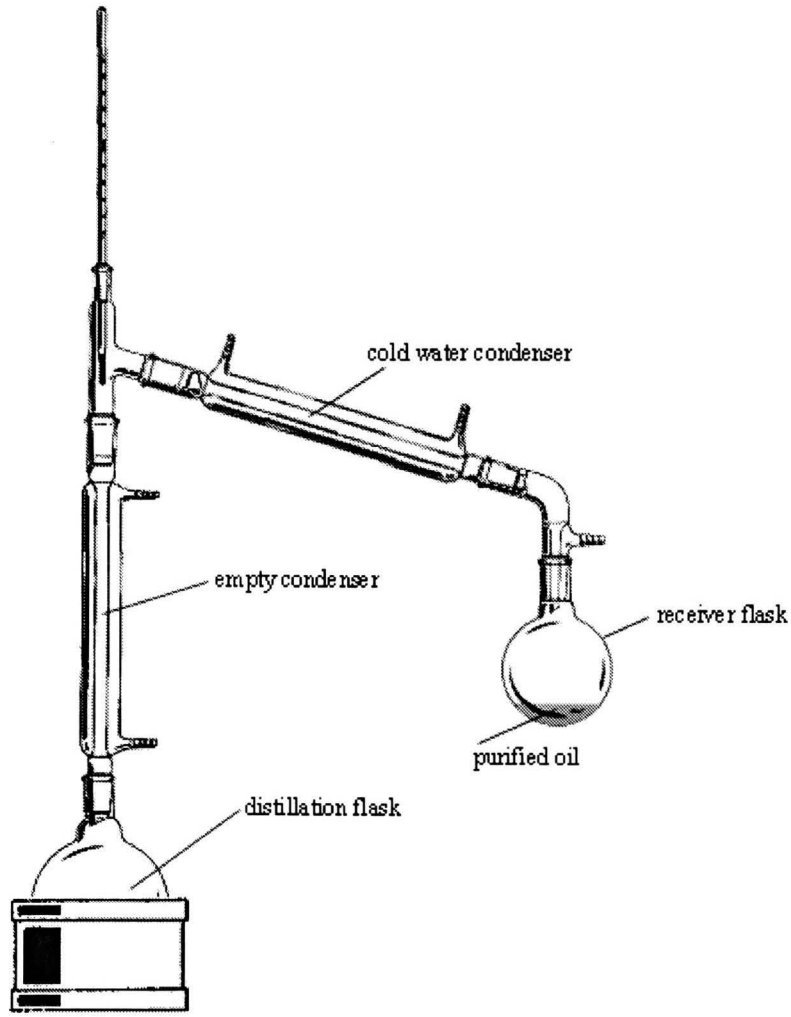


Figure 050. Fractional distillation apparatus for purification of asarone

Step 2: Preparation of the nitro intermediate

Into a suitable 3-neck flask, equipped with addition funnel, motorized stirrer or other stirring means, and thermometer, place 20 grams of the fractionally distilled asarone oil (obtained in step 1), followed by 200 milliliters of diethyl ether, then followed by 100 grams of sodium nitrite, and then finally followed by 250 milliliters of water. Thereafter, rapidly stir the mixture for about 30 minutes at room temperature to form a uniform mixture. Then place 160 milliliters of a 25% sulfuric acid solution into the addition funnel, and then slowly add, drop-wise, this dilute sulfuric acid solution to the asarone mixture over a period of about 1 hour, while rapidly stirring the asarone mixture and maintaining it temperature around 25 to 30 celsius at all times. After the addition of the dilute sulfuric acid, continue to stir the reaction mixture at room temperature overnight. The next day, allow the reaction mixture to stand at room temperature for about 2 hours. After 2 hours, filter-off the precipitated crystals, and then wash these filtered-off crystals with two 50-millilter portions of ice cold water, and then followed by one 50-milliliter portion of ice-cold diethyl ether. After the washings, vacuum dry or air-dry the crystals. Now, dissolve these washed dried crystals into an alkaline alcohol solution, prepared by adding and dissolving 10 grams of anhydrous sodium carbonate into 100 milliliters of 95% ethyl alcohol. Note: to speed up the dissolving time, gently heat the entire alcohol mixture to about 30 Celsius, and rapidly stir the entire mixture for about 30 minutes, or until all the crystals dissolve. Once all the crystals dissolve, remove the heat source, and allow the alcohol mixture to cool to room temperature. Note: during this brief and mild cool down period, moderately stir the alcohol mixture. Once the alcohol mixture reaches a temperature of about room temperature, allow the entire alcohol mixture to stand for about 15 minutes, and then pour this entire alcohol mixture into a large clean beaker, and then add in 300 grams of crushed ice. Immediately after adding in the ice, add in 200 milliliters of a 10% hydrochloric acid solution, and then stir the entire mixture until all the ice melts. When all the ice has melted, place the mixture into an ice bath, and chill to about 0 Celsius for about 1 hour with no stirring. After 1 hour, filter-off the precipitated crystals, and then vacuum dry or air-dry them.

Step 3: Preparation of 2,4,5-trimethoxyphenyl-2-propanone

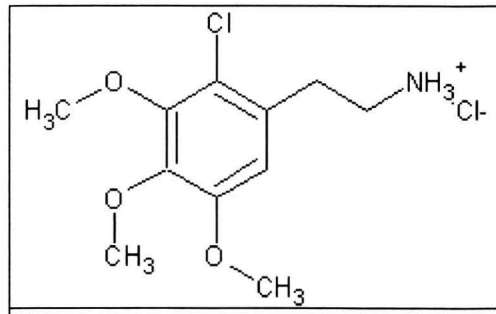
Into a standard reflux apparatus, equipped with motorized stirrer or other stirring means, and addition funnel, place 12.5 grams of the product obtained in step 2, followed by 10 grams of iron fillings, followed by 200 milligrams of anhydrous ferric chloride, and then followed by 20 milliliters of toluene. Thereafter, place 20 milliliters of 35 to 38% hydrochloric acid (muriatic acid will work) into the addition funnel. Then reflux the contents in the reflux flask to about 110 Celsius. When the contents in the reflux flask begin to reflux at about 110 Celsius, slowly add in, drop-wise, the concentrated hydrochloric acid (contained in the addition funnel), over a period of about 50 minutes while rapidly stirring the reaction mixture. After the addition of the hydrochloric acid, continue to reflux the reaction mixture for about 20 minutes with rapid stiring. After the additional 20 minutes, remove the heat source, and allow the reaction mixture to cool to room temperature. Thereafter, pour the entire reaction mixture into a clean large beaker, and then add in 400 milliliters of cold water, and then stir the entire diluted reaction mixture for about 30 minutes. After stirring for 30 minutes, extract the entire diluted reaction mixture with four 100-mililiter portions of diethyl ether, and after the extraction process, combine all ether portions (if not already done so), and then dry this combined ether portion by adding to it, 10 grams of anhydrous magnesium sulfate. Note: after each extraction process, the ether will be the upper layer each time. After adding in the anhydrous magnesium sulfate, stir the entire mixture for about 10 minutes (to absorb moisture), and then filter-off the magnesium sulfate. Finally, place this filtered ether mixture into a distillation apparatus, and distill-off the ether at 40 Celsius. When no more ether passes over or is collected, stop the distillation process, and then recover the left over remaining residue (after it has cooled). Then set this recovered residue aside for step 4.

Step 4: Preparation of TMA2

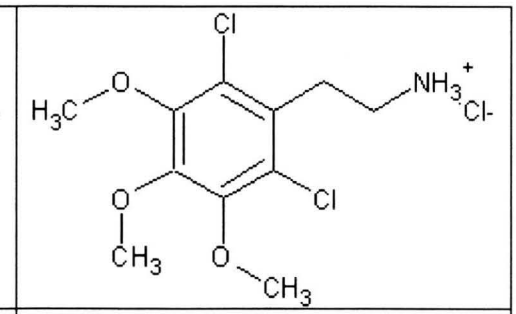
Into a suitable distillation apparatus, equipped with addition funnel, and cold-water condenser fitted with a receiver flask, place 6.6 grams of the product obtained in step 3, followed by 6 milliliters of formamide, followed by 1 milliliter of 90% formic acid. Thereafter, slowly heat this mixture to about 140 Celsius, and heat the mixture at this temperature for about 5 hours. Note: during the distillation process, a small amount of water will slowly distil over. Second note: during the distillation process, add in 1 milliliter of 90% formic acid every 30 minutes during the distillation process. This formic acid can be placed into the addition funnel, and then dripped in (1 milliliter at a time) every 30 minutes as just described. During the distillation process, carefully monitor the reaction mixture and keep its temperature around 140 Celsius at all times. Sometimes, to keep the reaction mixture at 140 Celsius during the 5-hour distillation process, the heat source may have to be removed occasionally to prevent excessive heat build-up caused by the chemical reaction. Also, monitor the gas evolution that will take place during the distillation process. If the gas evolution becomes vigorous or violent, immediately remove the heat source temporarily. After heating and distilling for about 5 hours, remove the heat source, and allow the reaction mixture to cool to room temperature. Afterwards, extract this entire reaction mixture with four 60-milliliter portions of toluene, and after the extraction process, combine all toluene portions (if not already done so), and then place this combined toluene portion into a distillation apparatus, and distill-off the toluene at 111 Celsius. Note: after each extraction, the toluene will be the upper layer each time; however, in most cases, the toluene can be simply decanted-off rather then collected by using a seperatory funnel. After distilling the toluene at 111 Celsius, and subsequently when no more toluene passes over or is collected, stop the distillation process, and then recover the left over remaining residue (after it has cooled). Now, place this collected left over residue into a clean reflux apparatus, and then add in 6 milliliters of 35 to 38% hydrochloric acid (muriatic acid will work), and then reflux this entire mixture at about 100 Celsius for about 90 minutes. After 90 minutes, remove the heat source, and allow the mixture to cool to room temperature. Then place this cooled mixture into a clean beaker, and then slowly and carefully add in, a sodium hydroxide solution prepared by adding 20 grams of sodium hydroxide into 100 milliliters of water. Note: sodium hydroxide generates excessive heat when dissolved in water, so allow this solution to cool to room temperature before using. After adding in the sodium hydroxide solution, rapidly stir the entire alkaline mixture for about 1 hour. Thereafter, extract this entire alkaline mixture with four 70-milliter portions of diethyl ether, and after the extraction process, combine all ether portions (if not already done so), and then dry this combined ether portion, by adding to it, 10 grams of anhydrous magnesium sulfate. Note: after each extraction, the ether will be the upper layer each time. After adding in the anhydrous magnesium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Finally, place this ether mixture into an ice bath, and then chill to 0 Celsius. Thereafter, bubble into this ether mixture 10 grams of dry hydrogen chloride gas (excess), and after the addition, allow the entire mixture to stand at 0 Celsius for about 1 hour. Thereafter, filter-off any precipitated crystals of the desired TMA2, and then wash these crystals with two 25-milliliter portions of diethyl ether, and then vacuum dry or air dry the crystals. The filtered ether mixture should then be concentrated by distillation at 40 Celsius until about 80% of the total volume has been reduced. Where upon, the resulting ether concentrate can then be filtered to recover some more precipitated crystals of the desired TMA2 product. These crystals should then be washed with one 25-milliliter portion of ether, and then vacuum dried or air-dried. The combined result will be a total of 6 grams of the desired product.

Note: other salts of the freebase TMA2 can be prepared in the usual manner, by replacing the hydrogen chloride gas with 98% sulfuric acid, pure tartaric acid, citric acid, or 80% phosphoric acid.

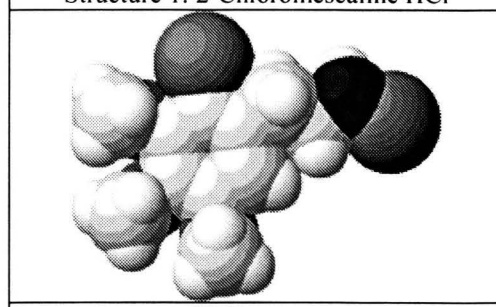
0037. ChloroMescaline. 2-Chloromescaline hydrochloride and 2,6-Dichloromescaline hydrochloride (DCM). *2-(2-chloro-3,4,5-trimethoxyphenyl)ethanamine hydrochloride and 2-(2,6-dichloro-3,4,5-trimethoxyphenyl)ethanamine hydrochloride (DCM)*



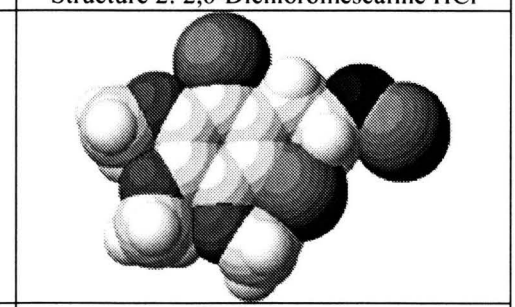
Structure 1: 2-Chloromescaline HCl



Structure 2: 2,6-Dichloromescaline HCl



3D Structure



3D Structure

2-chloromescaline hydrochloride and 2,6-dichloromescaline hydrochloride form colorless to white, to off-white crystals, which may be colored light yellow to light amber brown when impure. The melting point of the mixed chloromescaline hydrochlorides ranges from 215 to 230 Celsius. The 2,6-dichloromescaline forms beautiful shiny white plates with a melting point of 227 Celsius. Both compounds are soluble in water, alcohol, ether, methylene chloride, and other organic solvents at elevated temperatures. The crystals are less soluble in ice-cold alcohol and/or ether. The chloromescaline hydrochlorides have only yet to be studied with any significance in the last 10 years. The predominant and more closely studied chloromescaline hydrochloride of interest, is the 2,6-dichloromescaline hydrochloride, also known as DCM. DCM is a powerful psychedelic amphetamine (also known as a "hyper" or "intensified" psychedelic/hallucinogenic amphetamine) with strong hallucinogenic and stimulant effects. The drug produces powerful emotional and psychological responses in the brain, which can lead to a wide variety of hallucinogenic activity. Many of these psychological activities include visions and flashbacks, awakenings, spiritual journeys and other emotional "trips", and strong hallucinations resulting in seeing strange objects, motions, and silly creatures—these strong hallucinations are often accompanied with humorous overtones resulting in powerful bursts of laughter and silly behaviors conducted by the user. Many of the emotional trips experienced, may last for up 6 hours or more, and are usually replaced with, or accompanied with powerful feelings of well-being, happiness, stupor, and strong humorous responses to everything in the surrounding world. As far as the emotional and other hallucinogenic activities of the drug are concerned, DCM is also capable of generating stimulation and strong doses of adrenaline like rushes. Some have described the drug as making them feel like they were strapped to the nose of a rocket. Whatever the effects may be, which may vary in individual users, this drug demonstrates by far the highest potential for use as a street drug then any other psychedelic amphetamine listed in this book—it is quite obvious that this drug would be heavily popular by high school kids, college students, partiers, and social aristocrats. Because of the remarkable and somewhat un-predictable effects produced by such halogen derivatives of psychedelic amphetamines, DCM has very well opened the door to a whole new avenue of psychedelic chemistry—as for many years, halogen derivatives of stimulants and psychedelic amphetamines were not significantly explored or synthesized with intent for them to be used as "hyper" or "intensified" psychedelic/hallucinogenic drugs. Overall, DCM produces an outstanding high, which encompasses the full spectrum of highs and effects that could not possibly be experienced by any straight amphetamine or hallucinogen compound such as methamphetamine or LSD alone. It should be noted that DCM is still an experimental drug, so exact dosage, rates of onset, effects, and possible side-effects have yet to be fully realized; as a result, dosage should be limited to a minimum, until other wise experimentations or consumption trials proves that dosage can be increased.

This substance is a controlled substance (psychedelic/hallucinogenic/stimulant) as listed in the US code of Federal regulations.

Toxicity: Low	**Rate of onset (average):** Below average—may take up to 2 hours for effects to kick-in, but they may appear within 60 to

	90 minutes.
Stimulation dosage (ingestion): 3 to 10 milligrams—current explored dosages include: 3, 5, and 10-milligram doses, with the most powerful effects achieved with the 10-milligram dosage. Note: this 10-milligram dose does not assume that higher dosages equal better effects.	**Duration of effects (average):** 13 to 22 hours
Stimulation dosage (inhalation): unknown	**Habit forming potential:** Mild
Stimulation dosage (injection): unknown	**Estimated value U.S. (based on procedure):** $20 to $22 per gram. **Note: street value for a $5.00 per 10 milligram hit = $500 per gram**

Procedure A: Preparation of chloromescaline hydrochlorides

Materials:

1. 8 grams of manganese dioxide, or 130 grams of a 5% sodium hypochlorite solution (Clorox bleach), or 7 grams of potassium permanganate, or 10 grams of calcium hypochlorite (65% available chlorine).	11. and/or 9 grams of manganese dioxide, or 155 grams of a 5% sodium hypochlorite solution (Clorox bleach), or 8.5 grams of potassium permanganate, or 13 grams of calcium hypochlorite (65% available chlorine).
2. 16 grams of 35 to 38% hydrochloric acid	12. and/or 133 grams of dry chloroform
3. 94 grams of dry chloroform	13. and/or 12 grams of mescaline (the hydrochloride) prepared in 0020. MESCALINE
4. 12 grams of mescaline (the hydrochloride) prepared in 0020. MESCALINE	14. and/or 5 grams of sodium hydroxide
5. 5 grams of sodium hydroxide	15. and/or 150 milliliters of diethyl ether or methylene chloride
6. 150 milliliters of diethyl ether or methylene chloride	16. and/or 10 grams of anhydrous magnesium sulfate
7. 10 grams of anhydrous magnesium sulfate	17. and/or 150 milliliters of dry chloroform
8. 150 milliliters of dry chloroform	18. and/or 42.5 grams of 95% ethyl alcohol
9. 50 milliliters of 95% ethyl alcohol	19. and/or 42.5 grams of diethyl ether
10. 50 milliliters of diethyl ether	

Summary: In procedure A, a mixture of 2-chlormescaline and 2,6-dichloromescaline is prepared by reacting a chlorine in chloroform solution with freebase mescaline. The entire reaction takes place in a chloroform solvent, and afterwards, the solvent is simply removed, and the left over residue is then dissolved into a suitable solvent mixture, and this solvent mixture is then evaporated in the usual manner. The result of the remaining residue will be predominantly a mixture of 2-chloromescaline hydrochloride and 2,6-dichloromesclaine hydrochloride. In procedure B, 2,6-dichloromescaline is the predominant product, and can be produced by reacting a chlorine in chloroform solution with freebase mescaline in an identical manner as for the preparation of the 2-chloromescaline hydrochloride and 2,6-dichloromescaline hydrochloride mixture; however, the reaction mixture is allowed to stand for a prolonged period of time, whereby the chloroform acts as a free radical, lending one its chlorines to the mescaline body. However, this conversion is very slow, and only produces small yields when free chlorine is absent. Nonetheless, the chloroform solvent does help promote the chlorination of the mescaline, and if chloroform is replaced by methylene chloride, the yields of reaction are reduced. After the prolonged reaction time, the reaction mixture is evaporated, and the left over residue is then taken-up into a solvent mixture. This solvent mixture is then chilled in a refrigerator for several days, and thereafter, filtered to recover the precipitated crystals of the desired 2,6-dichloromescaline hydrochloride. These crystals can then be purified by recrystallization from a similar solvent mixture.

Hazards: Use care when handling strong oxidizers like manganese dioxide, potassium permanganate, sodium hypochlorite, and calcium hypochlorite, as they are all highly reactive and capable of reacting with many organic substances. Mixtures of strong oxidizers and combustible materials can ignite and burn violently. Wear gloves when handling hydrochloric acid, and sodium hydroxide, as they both can cause skin irritation. Use proper ventilation when handling diethyl ether, and extinguish all flames before using. Diethyl ether is capable of forming explosive mixtures with air, so use care. Use proper ventilation and avoid inhalation of chloroform vapors.

Procedure:

Personnel notes for procedure A: Chloromescaline hydrochlorides

Procedure A: Preparation of mixed 2-chloromescaline hydrochloride (23%) and 2,6-dichloromescaline hydrochloride (56%). The remainder is unreacted mescaline (21%).

Set-up the chlorine generator as illustrated below, and then bubble the chlorine gas into the chloroform (as illustrated), to produce a 5% chlorine solution in chloroform. To generate the necessary amount of chlorine gas (5.67 grams), place into the reaction flask, 8 grams of manganese dioxide, or 130 grams of a 5% sodium hypochlorite solution (Clorox bleach), or 7 grams of potassium permanganate, or 10 grams of calcium hypochlorite (65% available chlorine). Then place into the addition funnel, a hydrochloric acid solution prepared by adding and dissolving 16 grams of 35 to 38% hydrochloric acid (muriatic acid of 31% will work) into 20 milliliters of water. Then drip this hydrochloric acid solution, onto the oxidizer contained in the reaction flask. The chlorine that is evolved is then carried over, and bubbled into the chloroform, whereby it dissolves forming the 5% chlorine in chloroform solution. To form this 5% chlorine in chloroform solution, place 94 grams of dry chloroform into the receiver flask.

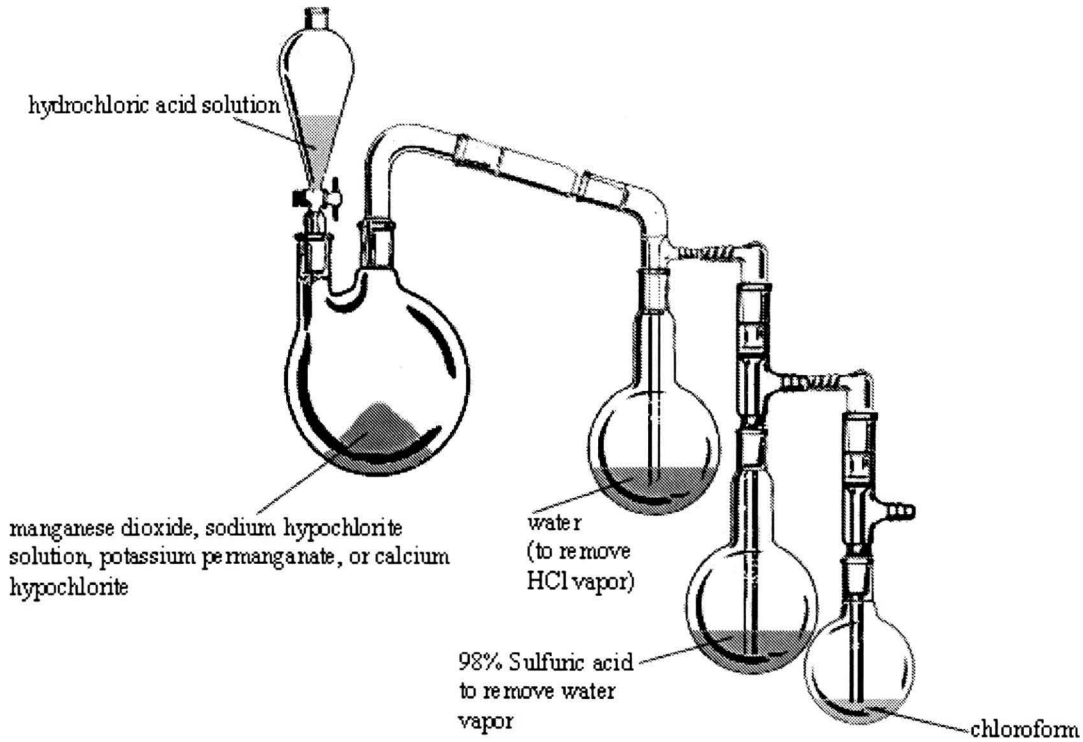


Figure 051. Set-up for the preparation of 5% chlorine in chloroform solution.

Once the 5% chlorine in chloroform solution has been prepared, set it aside for just a moment, and then place 12 grams of mescaline (the hydrochloride) prepared in 0020. MESCALINE into a suitable sized clean beaker, and then add in, 50 grams of a sodium hydroxide solution prepared by adding and dissolving 5 grams of sodium hydroxide into 45 milliliters of water. Note: sodium hydroxide generates excessive heat when dissolved in water, so allow the solution to cool to room temperature before using. After adding in the sodium hydroxide solution, stir the entire mixture for about 30 minutes. Thereafter, extract this entire mixture with three 50-milliter portions of diethyl ether or methylene chloride, and after the extraction process, combine all ether or methylene chloride portions (if not already done so), and then dry this combined ether or methylene chloride portion by adding to it, 10 grams of anhydrous magnesium sulfate. Note: after each extraction, the ether will be the upper layer each time, or if using methylene chloride, the methylene chloride will be the lower layer each time. After adding in the anhydrous magnesium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Finally, place the filtered ether or methylene chloride portion into a distillation apparatus, and distill-off the ether or methylene chloride at 40 Celsius. When no more ether or methylene chloride passes over or is collected, stop the distillation process, and then recover the left over resinous material (after it has cooled). Note: this resinous material will be composed of the freebase mescaline.

Now, dissolve this recovered left over resinous material (the freebase mescaline) into 150 milliliters of dry chloroform, and then stir the entire mixture for about 30 minutes—thereafter, quickly filter this mixture to remove any potential insoluble

materials (if any). Then place this filtered chloroform solution into a suitable sized flask or beaker (equipped with motorized stirrer or other stirring means), and then add in, the 5% chlorine in chloroform solution (previously prepared), over a period of about 5 to 10 minutes. Note: during the addition of the 5% chlorine in chloroform solution, rapidly stir the freebase mescaline mixture, and maintain its temperature around room temperature at all times. Second note: avoid direct contact of the reaction mixture and the 5% chlorine in chloroform solution with direct sunlight, or other sources of UV light, including lasers, laser pens, halogen lights, and/or other strong light sources. After the addition of the 5% chlorine in chloroform solution, rapidly stir the entire reaction mixture at room temperature for about 3 hours, and thereafter, place the entire reaction mixture into a distillation apparatus, and distill-off the chloroform at 62 Celsius. When no more chloroform passes over or is collected, stop the distillation process, and then recover the left over remaining residue (after it has cooled). Finally, dissolve this left over recovered residue into 100 milliliters of a warm solvent mixture prepared by adding and mixing 50 milliliters of 95% ethyl alcohol with 50 milliliters of diethyl ether (this solvent mixture can be pre-heated to about 40 Celsius prior to use). Thereafter, stir the entire mixture for about 30 minutes, and then filter-off any insoluble materials (if any). Then place this solvent mixture into a distillation apparatus, and distill-off both solvents by distillation at 78 Celsius. When no more ethyl alcohol or ether passes over or is collected, stop the distillation process, and then recover the left over remaining residue (after it has cooled). The left over residue will contain the mixed chloromescaline hydrochlorides. Note: there are numerous modifications that can be made to this process, and those who are trained should feel free to explore their own modifications.

Procedure B: Preparation of 2,6-dichloromescaline hydrochloride (95%+)

Set-up the chlorine generator as illustrated in procedure A, and then bubble the chlorine gas into the chloroform (as illustrated), to produce a 5% chlorine solution in chloroform. To generate the necessary amount of chlorine gas (6.9 grams), place into the reaction flask, 9 grams of manganese dioxide, or 155 grams of a 5% sodium hypochlorite solution (Clorox bleach), or 8.5 grams of potassium permanganate, or 13 grams of calcium hypochlorite (65% available chlorine). Then place into the addition funnel, a hydrochloric acid solution prepared by adding and dissolving 20 grams of 35 to 38% hydrochloric acid (muriatic acid of 31% will work) into 25 milliliters of water. Then drip this hydrochloric acid solution, onto the oxidizer contained in the reaction flask. The chlorine that is evolved is then carried over, and bubbled into the chloroform, whereby it dissolves forming the 5% chlorine in chloroform solution. To form this 5% chlorine in chloroform solution, place 133 grams of dry chloroform into the receiver flask.

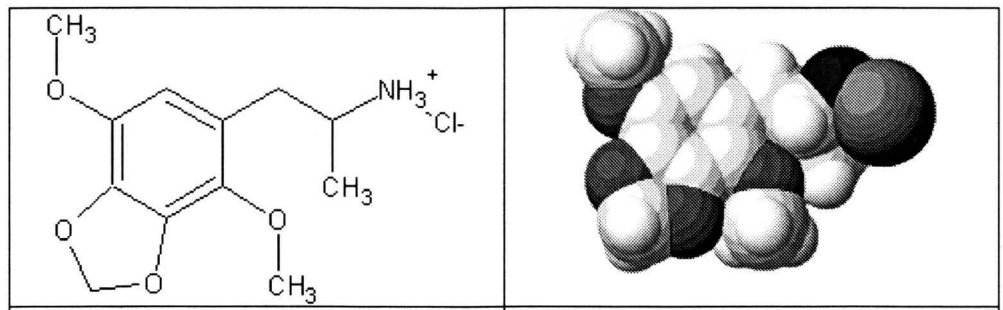
(Just as in procedure A): Once the 5% chlorine in chloroform solution has been prepared, set it aside for just a moment, and then place 12 grams of mescaline (the hydrochloride) prepared in 0020. MESCALINE into a suitable sized clean beaker, and then add in, 50 grams of a sodium hydroxide solution prepared by adding and dissolving 5 grams of sodium hydroxide into 45 milliliters of water. Note: sodium hydroxide generates excessive heat when dissolved in water, so allow the solution to cool to room temperature before using. After adding in the sodium hydroxide solution, stir the entire mixture for about 30 minutes. Thereafter, extract this entire mixture with three 50-milliter portions of diethyl ether or methylene chloride, and after the extraction process, combine all ether or methylene chloride portions (if not already done so), and then dry this combined ether or methylene chloride portion by adding to it, 10 grams of anhydrous magnesium sulfate. Note: after each extraction, the ether will be the upper layer each time, or if using methylene chloride, the methylene chloride will be the lower layer each time. After adding in the anhydrous magnesium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Finally, place the filtered ether or methylene chloride portion into a distillation apparatus, and distill-off the ether or methylene chloride at 40 Celsius. When no more ether or methylene chloride passes over or is collected, stop the distillation process, and then recover the left over resinous material (after it has cooled). Note: this resinous material will be composed of the freebase mescaline.

Now, dissolve this recovered left over resinous material (the freebase mescaline) into 150 milliliters of dry chloroform, and then stir the entire mixture for about 30 minutes—thereafter, quickly filter this mixture to remove any potential insoluble materials (if any). Then place this filtered chloroform solution into a suitable sized flask or beaker (equipped with motorized stirrer or other stirring means), and then add in, the 5% chlorine in chloroform solution (previously prepared), over a period of about 10 to 15 minutes. Note: during the addition of the 5% chlorine in chloroform solution, rapidly stir the freebase mescaline mixture, and maintain its temperature around room temperature at all times. Second note: avoid direct contact of the reaction mixture and the 5% chlorine in chloroform solution with direct sunlight, or other sources of UV light, including lasers, laser pens, halogen lights, and/or other strong light sources. After the addition of the 5% chlorine in chloroform solution, moderately stir the entire reaction mixture at room temperature for about 24 hours, and thereafter, place the entire reaction mixture into a distillation apparatus, and distill-off the chloroform at 62 Celsius. When no more chloroform passes over or is collected, stop the distillation process, and then recover the left over remaining residue (after it has cooled). Finally, dissolve this left over recovered residue into 35 grams of a warm solvent mixture prepared by adding and mixing 17.5 grams of 95% ethyl alcohol with 17.5 grams of diethyl ether (this solvent mixture can be pre-heated to about 40 Celsius prior to use). Thereafter, stir the entire mixture for about 30 minutes, and then quickly filter-off any insoluble materials (if any). Then place this solvent mixture into a stoppered flask, and then place this flask into a refrigerator, and allow it to stand for 2 or 3 days at a temperature of around 10 to 5 Celsius. After 2 or 3 days, remove the flask from the refrigerator, and then filter-off the precipitated crystals.

Now, recrystallize these dried filtered-off crystals from a similar solvent mixture prepared by adding and mixing 25 milliliters of 95% ethyl alcohol into 25 milliliters of diethyl ether. After the recrystallization process, vacuum dry or air-dry the filtered-off crystals. These refined crystals will be composed of the desired 2,6-dichloromescaline hydrochloride with a purity of about 95%+. The left over mother liquor, after the recrystallization process, will contain small amounts of unreacted mescaline hydrochloride, and 2-chloromescaline hydrochloride. Note: there are numerous modifications that can be made to this process, and those who are trained should feel free to explore their own modifications.

Note: other salts of the freebase chloromescaline hydrochlorides, especially of DCM, can be prepared by first, reacting the chloromescaline hydrochloride with a sodium carbonate solution, and then extracting the freebase containing alkaline mixture with three portions of ether or methylene chloride. The resulting combined ether or methylene chloride portions can then be treated with the desired acid, to form the desired salt. After reaction with the corresponding acid, the entire solvent mixture should be evaporated to dryness, and the resulting left over residue then dissolved into a minimal amount of an ethyl alcohol/ether mixture (50:50) as similar to the ethyl alcohol/ether solvent mixture discussed in the above process. This ethyl alcohol/ether mixture can then be quickly filtered, to remove any insoluble materials, and then placed into a refrigerator or freezer and allowed to chill for several days. After several days, the solvent mixture can be filtered to recover the precipitated crystals of the desired product. These crystals can then be recrystallized from an ethyl alcohol/ether mixture as similar to the above process.

0038. DMMDA. 2,5-Dimethoxy-3,4-methylenedioxyamphetamine hydrochloride. *1-(4,7-dimethoxy-1,3-benzodioxol-5-yl)propan-2-amine hydrochloride*



Structure | 3D Model

DMMDA forms white to slightly off-white to pale white crystals with a melting point of 175 Celsius. The impure crystals will have a melting point ranging from 164 to 176 Celsius. DMMDA is not your usual psychedelic amphetamine, and its main focus of effects resemble that of LSD. The hallucinogenic nature of the drug is somewhat related to LSD and other LSD derivatives with powerful emphasis's on images, pictures, statues, and motionless objects. The emphasis on images, pictures and motionless objects is accompanied with emotional feelings that usually connect with the images/pictures and/or motionless objects in the surrounding world in some spiritual fashion—meaning emotional thoughts and feelings are greatly enhanced by looking at images, pictures, statues, and motionless objects, resulting in spiritual overtones—not necessarily of "God" or "religious" like overtones. The hallucinogenic effects are not necessarily confined to emotional enchantments to imagery, and the effects also include the typical enhancements to sight, sound, color, and touch. Even though this drug produces similar effects as to LSD, it is by no means a suitable substitute for the famed LSD. Along with its hallucinogenic nature, DMMDA also produces (in conjunction with the hallucinogenic activity), a relatively good feeling high with a controllable and good natured intoxification that is similar to an alcohol intoxification—minus any dizziness, stupor, clumsiness, or other "drunk" like actions. Overall, DMMDA generates a good feeling intoxification, along with hallucinogenic effects that greatly enhance the surrounding world—leading to strange visual overtones to images, signs, motionless objects, moving objects, colors, lights, fixed objects, or any object of matter.

This substance is a controlled substance (hallucinogenic/psychedelic amphetamine) as listed in the US code of Federal regulations.

Toxicity: Mild—nausea may happen after 1 hour with small doses (doses below 50 milligrams)	**Rate of onset (average):** Average—may take up to 90 minutes for true effects to be realized
Stimulation dosage (ingestion): 30 to 75 milligrams—average dose is 75 milligrams	**Duration of effects (average):** 6 to 8 hours
Stimulation dosage (inhalation): unknown	**Habit forming potential:** Low
Stimulation dosage (injection): unknown	**Estimated value U.S. (based on procedure):** $25 to $27 per gram

Procedure A: Preparation of DMMDA

Materials:

1a. 150 grams of commercially available "Oil of Parsley"	16. 30 milliliters of pre-heated 95% ethyl alcohol (heated to about 50 celsius prior to use)
2a. 150 milliliters of diethyl ether	17. 15 milliliters of boiling hexane
3b. or 1 kilogram of finely ground parsley seeds (regular parsley)	18. 3.2 grams of pyridine
4b. or 75 milliliters of diethyl ether	19. 63 milliliters of acetone
5c. or 1 kilogram of parsley seeds	20. 6.5 grams of tetranitromethane
6c. or 500 milliliters of 95% ethyl alcohol	21. 3.1 grams of lithium aluminum hydride
7c. or 300 milliliters of diethyl ether	22. 500 milliliters of dry tetrahydrofuran
8c. or 20 grams of anhydrous sodium sulfate	23. 150 milliliters of a 5% sulfuric acid solution
9c. or 50 grams of sodium carbonate	24. 20 grams of anhydrous sodium carbonate
10c. or 10 grams of lead-II-oxide	25. 3 grams of picric acid
11d. or 1 kilogram of finely chopped-up parsley root	26. 50 milliliters of boiling 95% ethyl alcohol
12d. or 150 milliliters of a 10% sodium hydroxide solution	27. 50 milliliters of a 10% sodium hydroxide solution
13d. or 150 milliliters of diethyl ether	28. 200 milliliters of diethyl ether
14d. or 10 grams of anhydrous sodium sulfate	29. 10 grams of anhydrous magnesium sulfate
15. 24.1 grams of potassium hydroxide	30. 10 grams of dry hydrogen chloride gas

Summary: DMMDA is prepared in a four-step process starting with the isolation of apiole from commercial oils, or directly from parsley seeds or parsley roots. Apiole is a colorless to white crystalline material with a melting point of 30 Celsius. The crystals readily melt to form an oil. Apiole is readily available by fractional distillation of commercial oil of parsley, or from steam distillation of parsley seeds or parsley root, followed by solvent treatment to recover the apiole. Apiole can also be obtained by solvent extraction of the seeds, and then removal of the apiole from impurities by extraction into ether. The ether is then removed in the usual manner, and the left over residue is then treated with lead oxide, and sodium carbonate in the presence of water. The treated residue is then extracted into ether, and the ether extract is then removed in the usual manner. However the method of isolation, the desired apiole is then converted into 2,5-dimethoxy-3,4-methylenedioxy-1-propenylbenzene by reaction with potassium hydroxide in the presence of ethyl alcohol. The reaction mixture is then refluxed for a specified amount of time, allowed to cool, and then treated with water to induce precipitation of the desired 2,5-dimethoxy-3,4-methylenedioxy-1-propenylbenzene. This desired intermediate is then purified by recrystallization from boiling hexane. The purified crystals of the 2,5-dimethoxy-3,4-methylenedioxy-1-propenylbenzene are then converted into the nitro intermediate, 1-(2,5-dimethoxy-3,4-methylenedioxyphenyl)-2-nitropropene by condensation with tetranitromethane at ice cold temperatures in the presence of pyridine and acetone. After the reaction, the mixture is treated with base, and then stirred to allow formation of yellow crystals. These yellow crystals are then filtered-off, washed, and then dried. The yellow crystals of the desired nitro intermediate are then converted into the final desired product of DMMDA by reduction with lithium aluminum hydride in the usual manner. After the initial reduction, the reaction mixture is treated with acid (to destroy the lithium salts), and then basified by the addition of sodium carbonate. Thereafter, the reaction mixture is quickly boiled, filtered, cooled, and then treated with a hot solution of picric acid. The resulting precipitated picric acid salt is then filtered-off, made basic with a strong alkaline solution, and the resulting alkaline mixture is then extracted with ether. The ether extract is then saturated with hydrogen chloride gas, whereupon the desired DMMDA product precipitates.

Hazards: Extinguish all flames before using diethyl ether and tetrahydrofuran, which is highly volatile and flammable, and capable of forming explosive mixtures with air. Extinguish all flames before using hexane and acetone, both of which are highly volatile and flammable as well. Wear gloves when handling potassium hydroxide, sodium hydroxide, and sulfuric acid, as they are capable of causing skin irritation. Lithium aluminum hydride is highly reactive, and should be kept away form moisture—wear gloves when handling, as skin irritation will result with bare hands. Tetranitromethane is flammable, and should be handed with care, as should picric acid—avoid contact of both these compounds with strong bases, and heavy metal salts to avoid formation of shock sensitive compounds.

Procedure:

Personnel notes for procedure A: DMMDA

Step 1: Isolation of apiole from commercial oil of parsley (method 1)

Set-up the vacuum fractional distillation apparatus as illustrated below, and then fractionally distil 150 grams of commercially available "Oil of Parsley", at 167 celsius under a vacuum of 27 millimeters of mercury. After the vacuum distillation process, remove the ice trap from the receiver flask, and then allow the receiver flask to warm to room temperature. Thereafter, gently warm the receiver flask using a small Bunsen flame or other means, and allow the crystallized apiole to liquefy into an oil. Then pour this oil into an amber glass bottle, and then store it in a refrigerator until use. Note: in some cases, the apiole can be obtained by allowing the commercial oil of parsley to stand in an ice bath for several hours (a freezer can be used as well). During the chilling process, crystals of apiole will slowly form. These crystals can then be filtered-off, and then dissolved into 150 milliliters of ether. The ether mixture should then be briefly stirred for about 30 minutes, and then filtered to remove any potential insoluble impurities (if any). Then place this filtered ether mixture into a distillation apparatus, and distill-off the ether at 40 Celsius. When all the ether has been removed, place the left over remaining oily residue of the apiole (after it has cooled) into a amber glass bottle and store in a refrigerator until use. Note: There are numerous modifications to this process, and those who are willing, should carryout any modifications that would seem fit.

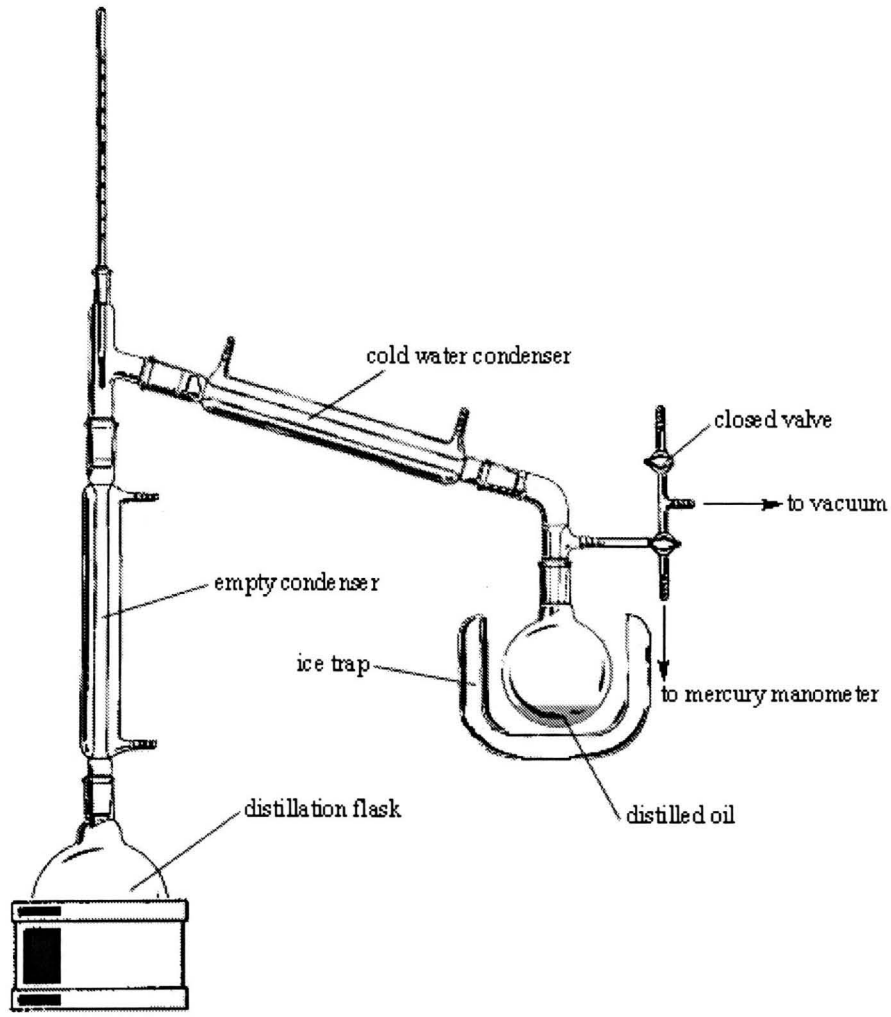


Figure 052. Vacuum fractional distillation set-up for the isolation of apiole from commercial oil of parsley.

Step 1: Isolation of apiole from oil of parsley obtained by steam distillation of parsley seeds (method 2)

Into a suitable steam distillation apparatus (as similar to 0036. TMA2, procedure A, step 1), place 1 kilogram of finely ground parsley seeds (regular parsley), and then add in 1500 milliliters of water. Note: the receiver flask used on the steam distillation apparatus, should be kept in a warm water bath to allow the volatized oil to remain as a liquid. Thereafter, steam distill the entire mixture at 100 Celsius for about 5 to 8 hours. Note: the exact amount of time may vary, and should be continued until no more oily resinous materials are collected in the receiver flask. When no more resinous oily material is seen collecting in the

receiver flask, stop the steam distillation process, and then pour the entire contents of the receiver flask into a seperatory funnel, and then allow the seperatory funnel to stand for about 1 hour (upright). Note: the seperatory funnel may need to be gently warmed using a free flame in order to keep the oil as a liquid. If the oily resinous material is allowed to cool, crystallization will result. After allowing the contents in the seperatory funnel to stand for about 1 hour, remove the lower oily layer (in some cases the oily layer may be the upper layer). After collecting the oily layer, place this oily layer into an ice bath (or use a freezer), and allow it to stand for several hours. After several hours, filter-off the crystallized apiole, and then dissolve this solid apiole into 75 milliliters of diethyl ether. Thereafter, briefly stir the ether mixture for about 30 minutes, and then filter-it to remove any insoluble impurities (if any). Thereafter, place this filtered ether mixture into a distillation apparatus, and distill-off the ether at 40 Celsius. When no more ether passes over or is collected, stop the distillation process, and then recover the left over remaining oily residue (before it cools). Then place this left over oily residue (composed primarily of the desired apiole) into an amber glass bottle, and store in a refrigerator until use. Note: There are numerous medications to this process, and those who are willing, should carryout any modifications that would seem fit.

Step 1: Isolation of apiole from parsley seeds via extraction (method 3)

Grind up 1 kilogram of parsley seeds until the seeds are of a finely ground nature. Then place the finely ground seeds into a large reflux apparatus (equipped with motorized stirrer or other stirring means), and then add in 500 milliliters of 95% ethyl alcohol. Thereafter, reflux the entire mixture at 78 Celsius for about 6 to 8 hours while moderately stirring the ethyl alcohol mixture. After refluxing for about 6 to 8 hours, remove the heat source, and then allow the entire alcohol mixture to cool to room temperature. Thereafter, filter the entire alcohol mixture to remove any insoluble materials, and then place this ethyl alcohol mixture into a distillation apparatus, and distill-off the ethyl alcohol until about 50% of its total volume has been reduced (about 250 milliliters of ethyl alcohol removed). Note: the recovered ethyl alcohol can be recycled if desired. When about 50% of the ethyl alcohol mixture has been removed, stop the distillation process, and then place the ethyl alcohol mixture into a suitable sized beaker (before it cools), and then allow it to cool to room temperature. Then, quickly filter this alcohol concentrated mixture to remove any potential insoluble impurities (if any). Now, extract this entire alcohol mixture with three 50-millilter portions of diethyl ether, and after the extraction process, combine all ether portions (if not already done so), and then dry this combined ether portion by adding to it, 10 grams of anhydrous sodium sulfate. Note: after each extraction, the ether will be the upper layer each time. After adding in the sodium sulfate, stir the entire ether mixture for about 10 minutes, and then filter-off the sodium sulfate. Then, place this filtered ether mixture into a distillation apparatus, and distill-off the ether at 40 Celsius. When no more ether passes over or is collected, stop the distillation process, and then recover the left over remaining oily residue (when it cools to about 40 Celsius). Then place this warm collected left over oily residue into a clean beaker, and then add in 50 milliliters of warm water, followed by 50 grams of sodium carbonate, and then followed by 10 grams of lead-II-oxide. Thereafter, rapidly blend this entire mixture for about 1 hour at a temperature of about 40 Celsius—a hot plate will be needed in order to keep the temperature of the mixture at about 4 Celsius. After rapidly stiring for about 1 hour, add in 50 milliliters of additional warm water, and then continue to stir the entire mixture at about 40 Celsius for an additional hour. Thereafter, filter the entire mixture through a layer of charcoal (place a bed of charcoal over the filter paper), before the mixtures temperature drops below 40 Celsius. After the filtration process, place the entire filtered mixture into a clean beaker. Now, extract this entire mixture with three 50-milliliter portions of diethyl ether, and after the extraction process, combine all ether portions (if not already done so), and then dry this combined ether portion, by adding to it, 10 grams of anhydrous sodium sulfate. Note: after each ether extraction, the ether will be the upper layer each time. After adding in the anhydrous sodium sulfate, stir the entire ether mixture for about 10 minutes, and then filter-off the sodium sulfate. Finally, place this entire ether mixture into a distillation apparatus, and distill-off the ether at 40 Celsius. When no more ether passes over or is collected, stop the distillation process, and then recover the left over remaining oily residue (before it cools to below 40 Celsius). Then place this warm collected left over residue (composed primarily of the desired apiole) into an amber glass bottle, and then store it in a refrigerator until use. Note: There are numerous medications to this process, and those who are willing, should carryout any modifications that would seem fit.

Step 1: Isolation of apiole by steam distillation of parsley root—essential oil of parsley (method 4)

Apiole can also be obtained by isolation from the essential oil of parsley, which is obtained by steam distillation of parsley root. To obtain apiole in this manner, place 1 kilogram of finely chopped-up parsley root, into a standard reflux apparatus, followed by 1500 milliliters of water, and then followed by 150 milliliters of a 10% sodium hydroxide solution. Thereafter, reflux the entire mixture for about 6 hours at 100 Celsius. After 6 hours, allow the entire mixture to cool to room temperature, and then pour the entire mixture (including any solid materials) into a standard steam distillation apparatus (as similar to 0036. TMA2, procedure A, step 1), and then steam distill the entire mixture at 100 Celsius for about 6 to 8 hours. Note the actual time may vary. Continue to steam distill the entire mixture until no more oily resinous material is seen collecting in the receiver flask. Note: the receiver flask on the steam distillation apparatus should be contained in a warm water bath, to prevent crystallization of any volatized oil. When no more oily resinous material is seen collecting in the receiver flask, stop the steam distillation process, and then pour all the contents of the receiver flask, into a clean beaker, and then extract this entire mixture with three 50-milliilter portions of diethyl ether. After the extraction process, combine all ether portions (if not already done

so), and then dry this combined ether portion, by adding to it, 10 grams of anhydrous sodium sulfate. Note: after each extraction, the ether will be the upper layer each time. After adding in the anhydrous sodium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the sodium sulfate. Finally, place this filtered ether mixture into a distillation apparatus, and distill-off the ether at 40 Celsius. When no more ether passes over or is collected, stop the distillation process, and recover the left over oily residue (before it cools below 40 Celsius). Then place this recovered oily residue (composed of primarily the desired apiole) into an amber glass bottle, and then store it in a refrigerator until use. Note: There are numerous medications to this process, and those who are willing, should carryout any modifications that would seem fit.

Step 2: Preparation of 2,5-dimethoxy-3,4-methylenedioxy-1-propenylbenzene

Into a standard reflux apparatus, equipped with motorized stirrer or other stirring means, place 10 grams of apiole (obtained in either method of the step 1), followed by 22 grams of potassium hydroxide, and then finally followed by 30 milliliters of pre-heated 95% ethyl alcohol (heated to about 50 celsius prior to use). Thereafter, reflux this entire mixture at about 79 Celsius for about 12 hours while rapidly stirring the mixture. After 12 hours, remove the heat source, and allow the refluxing mixture to cool to room temperature. Then pour this entire reaction mixture into a suitable sized beaker, and then relatively fast, but not to slow, add in, 50 to 100 milliliters of ice-cold water. Note: during the addition of the water, the desired product will crystallize out. During the addition of the ice-cold water, rapidly stir the mixture. Keep adding in the ice-cold water until no more crystals form. Once all the crystals have crystallized out of the reaction mixture, filter-off the precipitated amber colored crystals, and then vacuum dry or air-dry these filtered-off crystals. Then dissolve these dried crystals into 15 milliliters of boiling hexane, and then quickly and rapidly stir the entire mixture for about 5 minutes, and the quickly filter the mixture to remove any insoluble impurities. Immediately thereafter, recrystallize the dissolved product from this boiling hexane mixture. Note: in many cases, the hot hexane mixture will give rise to crystallization inside the filtering flask after the filtration. If this happens, never mind it, and allow the hexane mixture to cool to room temperature—this should be avoided however, and the filtered hot hexane mixture should be transferred to a beaker before it is allowed to cool, because the precipitated crystals may be difficult to remove from the filtering flask. After the recrystallization process, vacuum dry or air-dry the collected filtered-off crystals. The result will be about 4.6 grams of the desired product as pale crystals.

Step 3: Preparation of 1-(2,5-dimethoxy-3,4-methylenedioxyphenyl)-2-nitropropene

Into a suitable sized breaker or flask (equipped with motorized stirrer or other stirring means), place 7.3 grams of the product obtained in step 2, followed by 3.2 grams of pyridine, and then followed by 38 milliliters of acetone. Then stir this entire mixture for about 30 minutes at room temperature to form a uniform mixture. Thereafter, place this mixture into an ice bath, and chill to about 0 Celsius. When its temperature reaches about 0 Celsius, carefully and slowly add in, 6.5 grams of tetranitromethane (see The Preparatory Manual of Explosives for its preparation). During the addition of the tetranitromethane, rapidly stir the entire reaction mixture for about 30 minutes. After the addition, continue to stir the entire reaction mixture at 0 Celsius for about 10 minutes. Then, prepare a potassium hydroxide solution by adding and dissolving 2.1 grams of potassium hydroxide into 38 milliliters of cold water. Note: potassium hydroxide generates excessive heat when dissolved in water, so allow the alkaline solution to cool before using. Then gradually add this potassium hydroxide solution to the reaction mixture over a period sufficient to keep the reaction mixtures temperature below 5 Celsius at all times. During the addition of the potassium hydroxide solution, rapidly stir the reaction mixture. After the addition of the potassium hydroxide solution, continue to stir the reaction mixture until yellow crystals precipitate (reduce the stirring speed to slow). Once yellow crystals precipitate, continue to stir on slow speed, for about 30 minutes to make sure all yellow crystals have precipitated. Thereafter, filter-off these yellowish crystals, then wash them with 50 milliliters of a 50% acetone solution (prepared by adding and mixing 25 milliliters of cold water with 25 milliliters of acetone). After washing the crystals, vacuum dry or air-dry them. The result will be about 6 grams of the desired nitro intermediate compound, with a melting point of about 111 Celsius.

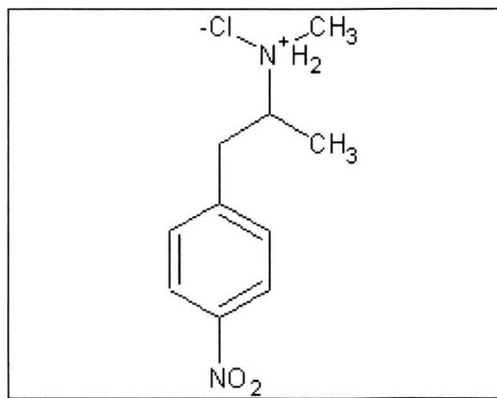
Step 4: Preparation of DMMDA

Into a standard reflux apparatus, equipped with motorized stirrer or other stirring means, and addition funnel, place 3.1 grams of lithium aluminum hydride, followed by 250 milliliters of dry tetrahydrofuran. Thereafter, begin a gentle reflux of this lithium aluminum hydride mixture by heating to 68 Celsius with moderate stirring. Note: keep a calcium chloride drying tube attached to the reflux condenser to exclude air. Then prepare a solution by adding and dissolving 3.7 grams of the product obtained in step 3 into 250 milliliters of dry tetrahydrofuran. Thereafter, place this solution into the addition funnel, and then slowly add it, drop-wise, over a period of 45 minutes. During this addition, rapidly stir the lithium aluminum hydride mixture and maintain it at reflux around 68 Celsius. After the addition, continue to reflux the lithium aluminum hydride (reaction mixture) for about 90 additional minutes with rapid stirring. After 90 minutes, remove the heat source, and allow the reaction mixture to cool to room temperature. Then pour the entire reaction mixture into a suitable sized beaker, and then carefully and slowly add in, 150 milliliters of a 5% sulfuric acid solution. During the addition of the dilute sulfuric acid solution, rapidly stir the reaction mixture. After the addition, carefully and slowly add in, 20 grams of anhydrous sodium carbonate. During the addition of the sodium carbonate, rapidly stir the reaction mixture. After the addition, briefly heat the entire reaction mixture to

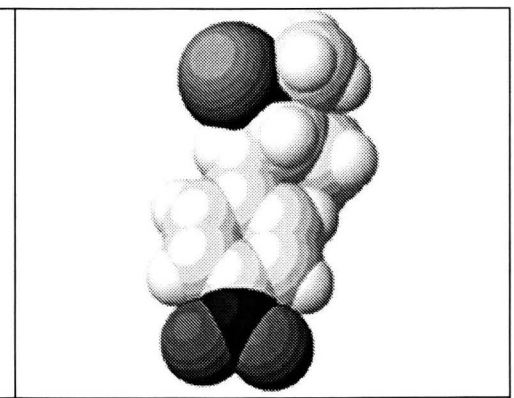
80 Celsius with stirring, and when the temperature of the reaction mixture reaches 80 Celsius, continue to stir and heat for about 10 minutes. Thereafter, remove the heat source, and allow the reaction mixture to cool to room temperature. Thereafter, filter the cooled reaction mixture to remove any potential insoluble impurities. Now, prepare a picric acid solution, by adding and dissolving 3 grams of picric acid (see The Preparatory Manual of Explosives for more info), into 50 milliliters of boiling 95% ethyl alcohol. As soon as the picric acid dissolves, add this picric acid/ethyl alcohol solution to the filtered reaction mixture, and then rapidly blend the entire mixture for about 10 minutes. After 10 minutes, filter-off the insoluble globs (which will be the insoluble picrate salt of DMMDA), and then wash these filtered-off globs of the picrate salt with three 50-millilter portions of ice cold water, and instead of drying these filtered-off globs, place these washed globs into a clean beaker, and then add in 50 milliliters of a 10% sodium hydroxide solution, and then rapidly stir the entire alkaline mixture for about 30 minutes. *Note: the picric acid can be recycled by recovering the sodium salt from the aqueous alkaline mixture (after the following extraction).* After stirring the alkaline mixture for about 30 minutes, extract this entire alkaline mixture with three 50-milliter portions of diethyl ether, and after the extraction process, combine all ether portions (if not already done so), and then dry this combined ether portion, by adding to it, 10 grams of anhydrous magnesium sulfate. Note: after each extraction, the ether will be the upper layer each time. After adding in the anhydrous magnesium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Finally, place this filtered ether portion into an ice bath, and chill to about 0 Celsius. Thereafter, bubble into this cold ether mixture, 10 grams of dry hydrogen chloride gas (excess). After the addition of the hydrogen chloride gas, stir the entire ether mixture for about 1 hour at 0 Celsius. Thereafter, filter-off the precipitated white crystals of the desired DMMDA, then wash them with one 50-millilter portion of diethyl ether, and then vacuum dry or air-dry the washed crystals. The result will be about 1.4 grams of the desired product.

Note: other salts of the freebase DMMDA can be prepared in the usual manner, by replacing the hydrogen chloride gas with 98% sulfuric acid, pure tartaric acid, citric acid, or 80% phosphoric acid.

0039. NitroMeth. Para-Nitro-phenyl-isopropylmethyl amine hydrochloride. *N-methyl-N-[1-methyl-2-(4-nitrophenyl)ethyl]amine hydrochloride*



Structure



3D Model

Nitrometh forms yellowish to pale yellow, to slightly colored transparent crystals with a melting point of 196 Celsius. The impure crystals may have a melting point of 188 Celsius. Nitrometh is slightly soluble in alcohol and methylene chloride, but only slightly soluble in water. Nitrometh is an interesting amphetamine with psychosomatic effects upon the brain. It produces central nervous system stimulation (similar to methamphetamine), with analgesic (pain killing) activity, mild psychedelic effects (similar to mescaline), and emotional tranquilizing effects—the latter is like meditation, whereby the user will be in a "catatonic" like state, but will have full mental awareness. The combined stimulation, mild psychedelic effects, and emotional tranquilizing effects makes nitrometh a vary strange compound. In this regard, the drug produces an excitation similar to methamphetamine, but with unusual sensations of relaxation with mild hallucinogenic mescaline like effects. Despite the unexpected sensations of relaxation, the drug is also capable of producing strong sensations of aggression and anger when users are provoked—meaning the users may feel strong anger and aggressive tendencies when provoked or "cornered". Rumors about this compound have stated that it (or similar compounds) where given to the last remaining German troops during WWII for their offensives during the "Battle of The Bulge", and in similar last ditch efforts against advancing Russian troops—this muffled and "hear-say" information has lead to the conclusion that this substance could be anticipated to be an "anger" or "aggressive" inducing drug—however, these anger and aggressive tendencies have proven to usually only exist when users are provoked, cornered, intimidated, or put into positions with threatening overtones. This drug can be classified as a psychedelic/tranquilizing/stimulant (shortened to "psychosomatic stimulant").

This substance is a controlled substance (psychosomatic stimulant) as listed in the US code of Federal regulations.

Toxicity: Quite low	**Rate of onset (average):** Moderate—effects are usually

	realized within 1 hour, depending on exact dosage
Stimulation dosage (ingestion): 150 to 250 milligrams—different doses may result in different effects. **Note: Persons new to this drug should start with 10-milligram doses, and work their way up.**	**Duration of effects (average):** 10 to 12 hours
Stimulation dosage (inhalation): unknown	**Habit forming potential:** Very low
Stimulation dosage (injection): unknown	**Estimated value U.S. (based on procedure):** unknown

Procedure A: Preparation of nitrometh

Materials:

1. 25 grams of methamphetamine hydrochloride (see 0009. Methamphetamine hydrochloride)	7. 100 milliliters of a 40% sodium hydroxide solution
2. 10 grams of sodium hydroxide	8. 300 milliliters of benzene
3. 300 milliliters of methylene chloride	9. 15 grams of anhydrous sodium sulfate
4. 10 grams of anhydrous magnesium sulfate	10. 75 milliliters of 95% ethyl alcohol
5. 12 milliliters of 98% sulfuric acid	11. 10 grams of dry hydrogen chloride gas
6. 13 milliliters of pre-chilled 99% nitric acid	

Summary: Nitrometh is prepared in a two-step process starting with the formation of freebase methamphetamine. The freebase compound is simply obtained by reacting the hydrochloride with a sodium hydroxide solution, and then extracting the resulting freebase containing mixture with methylene chloride. The methylene chloride extract is then evaporated, and the left over oil is then recovered. The freebase methamphetamine oil is then converted into the desired nitrometh, by reaction with highly concentrated nitric acid in the presence of sulfuric acid. After the reaction, the mixture is then drowned into ice water, and then resulting mixture is then extracted with benzene. The benzene extracts are then combined, dried, and then evaporated to leave behind a oily residue of the impure product This oily impure product is then dissolved into ethyl alcohol, the resulting alcohol solution is then filtered, and then treated with hydrogen chloride gas in the usual manner. The resulting acidified alcohol mixture is then allowed to stand for several hours, where upon the crystals of the nitrometh precipitate in the usual manner. For similar information, or for references regarding this process, see Serial number: 547,446 filed May 4th, 1966 to Zoltan Ecsery, Jozsef Knoll, Iidiko Kosa, Iidiko Sandor, Eva Somfai, and Sandor Totok, all of Budapest Hungary. Assigned to: Chinoin Gyogyszer es Vegyeszeti Termekek Byara RT.

Hazards: Use maximum ventilation when handling diethyl ether, which is highly flammable and capable of forming explosive mixtures with air. Wear gloves when handling sulfuric acid, sodium hydroxide, and hydrogen chloride—use proper ventilation when handling hydrogen chloride, which is highly irritating to the nose and throat. Use extreme care when handling 99% nitric acid, and use proper ventilation. 99% Nitric acid generates highly toxic fumes of nitrogen oxides, so use caution. Use proper ventilation when handling benzene, and avoid inhalation of the vapors—benzene is a suspected carcinogen.

Procedure:

Personnel notes for procedure A: Nitrometh

Step 1: Preparation of freebase methamphetamine

Into a suitable sized beaker, add in 25 grams of methamphetamine hydrochloride (see 0009. Methamphetamine hydrochloride), followed by a sodium hydroxide solution prepared by adding and dissolving 10 grams of sodium hydroxide (excess) into 75 milliliters of water. Note: sodium hydroxide generates excessive heat when dissolved in water, so allow the alkaline solution to cool before using. After adding in the sodium hydroxide solution, rapidly stir the entire mixture for about 30 minutes at room temperature. Thereafter, extract this entire alkaline mixture with three 100-milliter portions of methylene chloride, and after the extraction process, combine all methylene chloride portions, if not already done so, and then dry this combined methylene chloride portion, by adding to it, 10 grams of anhydrous magnesium sulfate. Note: after each extraction, the methylene chloride will be the lower layer each time. After adding in the anhydrous magnesium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Finally, place this filtered combined methylene chloride portion into a distillation apparatus, and distil-off the methylene chloride at 40 Celsius. When no more methylene chloride passes over or is

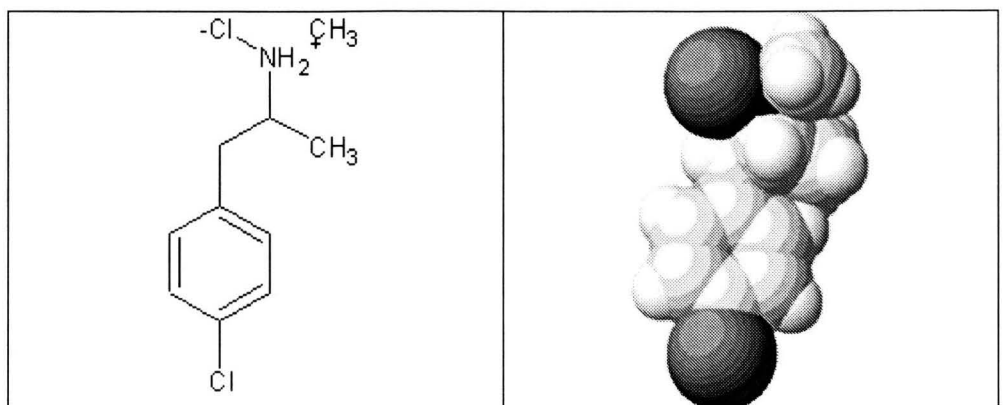
collected, stop the distillation process, and the recover the left over oil (after it has cooled). Then place this left over oil aside for step 2.

Step 2: Preparation of nitrometh

Into a suitable flask, equipped with motorized stirrer or other stirring means, thermometer, and addition funnel, place 8 milliliters of ice-cold water, followed by 12 milliliters of 98% sulfuric acid. Thereafter, allow this acid solution to cool to room temperature before proceeding. Afterwards, slowly add to this cooled sulfuric acid solution, in small portions at a time, 17 grams of the freebase methamphetamine (obtained in step 1), and during the addition, rapidly stir the sulfuric acid solution. After the addition, continue to stir the reaction mixture for about 10 minutes at room temperature, and then place 13 milliliters of pre-chilled 99% nitric acid (pre-chilled to about 10 celsius) into the addition funnel. Now, slowly add, drop-wise, this chilled 99% nitric acid, to the reaction mixture over a period of time sufficient to keep the reaction mixtures temperature below 35 Celsius at all times. Note: a cold-water bath may or may not be needed during the addition of the nitric acid, and the temperature of the reaction mixture should be carefully monitored all throughout the addition of the nitric acid. After the addition of the nitric acid, continue to stir the reaction mixture at about 25 to 30 Celsius for 2 hours. Thereafter, pour the entire reaction mixture into 200 milliliters of ice water, and then stir the resulting icy mixture for about 10 minutes. After about 10 minutes, slowly and carefully add in, 100 milliliters of a 40% sodium hydroxide solution. During the addition of the sodium hydroxide solution, rapidly stir the entire mixture. After the addition of the sodium hydroxide solution, rapidly stir the entire mixture for about 1 hour at room temperature. Then, extract this entire mixture with three 100-milliter portions of benzene, and after the extraction process, combine all benzene extracts (if not already done so), and then dry this combined benzene portion, by adding to it, 15 grams of anhydrous sodium sulfate. Note: after each extraction, the benzene will be the upper layer each time. After adding in the anhydrous sodium sulfate, stir the entire benzene portion for about 10 minutes, and then filter-off the sodium sulfate. Now, place this filtered benzene portion into a distillation apparatus, and distill-off the benzene at 80 Celsius. When no more benzene passes over or is collected, stop the distillation process, and then recover the left over remaining oily residue (after it has cooled). Now, dissolve this left over oily residue into 50 milliliters of 95% ethyl alcohol, and then briefly stir the entire mixture for about 30 minutes—thereafter filter the alcohol mixture to remove any potential insoluble materials (if any). Thereafter, bubble into this alcohol mixture, 10 grams of dry hydrogen chloride gas (excess). Finally, after the addition of the hydrogen chloride gas, place the alcohol mixture into an ice bath, and allow it to stand at 0 Celsius for about 6 hours. After 6 hours, filter-off the precipitated crystals of the desired nitrometh, and then wash these crystals with one 25-milliter portion of pre-chilled 95% ethyl alcohol (pre-chilled to about 0 Celsius), and then vacuum dry or air dry the crystals. Note: the final product will have small amounts of the ortho isomer, but this is not a concern.

Note: As usual, other salts of the freebase nitrometh can be obtained by replacing the hydrogen chloride gas with the desired acid—the exact amount of the desired acid does not have to be exact, and an excess is always good.

0040. Chlorometh. Chlorinated Methamphetamine. para-Chloro-phenylisopropyl methylamine hydrochloride. *N-[2-(4-chlorophenyl)-1-methylethyl]-N-methylamine hydrochloride*



Structure	3D Model

Chlorometh forms colorless to transparent crystals with a melting point of139 Celsius. The impure crystals may be off-white to slightly yellow or brown, with a melting point ranging from 135 to 142 Celsius. The crystals are soluble in water, alcohol, and slightly soluble in ether. Solubility in alcohol and ether is decreased with decreasing temperature. Chlorometh is another

interesting derivative of methamphetamine with analgesic, tranquilizing, and hallucinogenic effects. Chlorometh does not show the usual stimulation that would be expected from halogen derivatives of amphetamines, and it is in fact, a potent psychedelic compound with hallucinogenic and pain killing activity. The drug is about 10 times more potent then mescaline in the field of dosage, and several times more potent in the field of hallucinogenic activity. The pain killing activity is of coarse no where near the effects of morphine, but it is capable of general anesthesia resulting in a good natured high (similar to the high produced by opiates), with tranquilizing secondary effects. Even though the drug is limited in the areas of stimulation, it is capable of producing general central nervous system stimulation resulting in mild bursts of energy, motivation, mental concentration, and loss of appetite—the mild bursts of energy and mental concentration may appear and disappear during the intoxification. The hallucinogenic effects are similar to mescaline in nature, but exact psychedelic effects may vary in users due the tranquilizing effect of the drug. Overall, the potency and good natured high produced by this drug makes it suitable for use in street consumption; however, it does demonstrate several side-effects that may or may not appear in users—these side effects include: tiredness or sleepiness, decreased blood pressure, and blood thinning (anti-coagulant).

This substance is a controlled substance (psychosomatic/analgesic amphetamine) as listed in the US code of Federal regulations.

Toxicity: Moderate	**Rate of onset (average):** Average
Stimulation dosage (ingestion): 30 to 75 milligrams—new users should start at 25 milligram dose	**Duration of effects (average):** 10 to 12 hours
Stimulation dosage (inhalation): unknown	**Habit forming potential:** Low
Stimulation dosage (injection): unknown	**Estimated value U.S. (based on procedure):** $24 to $26 per gram

Procedure A: Preparation of chlorometh

Materials:

1. 14.5 grams of nitrometh (see 0039. Nitrometh)	9. 15 grams of sodium chloride
2. 375 milliliters of 95% ethyl alcohol	10. 6 to 10 grams of sulfur dioxide gas
3. 1.4 grams of a palladium on charcoal catalyst (average palladium content)	11. 20 milliliters of 35 to 38% hydrochloric acid
4. 500 milligrams of hydrogen gas	12. 5 grams of sodium nitrite
5. 20 grams of sodium hydroxide	13. 20 milliliters of a 28 to 30% ammonia solution
6. 830 milliliters of benzene	14. 10 grams of hydrogen chloride gas
7. 40 grams of anhydrous sodium sulfate	15. 100 milliliters of diethyl ether
8. 15 grams of anhydrous cupric sulfate	

Summary: Chlorometh is readily obtained in a two-step process starting with the formation of para-amino-phenylisopropyl methylamine. This amino intermediate is readily prepared by reducing nitrometh with hydrogen in the presence of a palladium/charcoal catalyst. The reaction is rather simple and mild, and afterwards, the reaction mixture is filtered, and then evaporated. The left over residue is then taken up into water, and the resulting aqueous mixture is then basified by the addition of sodium hydroxide. The basified mixture is then extracted with benzene, and the resulting benzene extracts are combined, dried, and then evaporated to leave behind a resinous oil, which is the desired amino intermediate. This intermediate is then converted into chlorometh by reaction with hydrochloric acid and nitrous acid (generated from sodium nitrite) in the presence of a sulfur dioxide containing mixture with copper sulfate and sodium chloride. The reaction is rather strange but nevertheless simple, and after the initial reaction, the entire mixture is refluxed. Afterwards, the reaction mixture is washed with benzene, and then basified by the addition of concentrated ammonia, followed by sodium hydroxide. The basified reaction mixture is then extracted with benzene, and the combined benzene extracts are then dried, and then evaporated to leave behind an oily residue. This oily residue is then taken-up into alcohol, whereby the desired chlorometh is then formed by addition of hydrogen chloride in the usual manner. The chlorometh is then collected by adding in ether and allowing the entire solvent mixture to stand for a prolonged period of time. The precipitated crystals thereafter, are then collected in the usual manner. For similar information, or for references regarding this process, see Serial number: 547,446 filed May 4th, 1966 to Zoltan Ecsery, Jozsef Knoll, Iidiko Kosa, Iidiko Sandor, Eva Somfai, and Sandor Totok, all of Budapest Hungary. Assigned to: Chinoin Gyogyszer es Vegyeszeti Termekek Byara RT.

Hazards: Extinguish all flames before using diethyl ether, which is highly flammable and capable of forming explosive mixtures with air—use caution. Hydrogen gas is highly flammable and explosive, so avoid all possible sources of ignition. Wear gloves when handling sodium hydroxide and hydrochloric acid, as they both can cause skin irritation. Use proper ventilation and avoid inhalation of sulfur dioxide gas, which is irritating and toxic. Use proper ventilation and avoid inhalation of benzene vapors—benzene is a suspected carcinogen. Use proper ventilation when handling concentrated ammonia, and avoid inhalation of the vapors.

Procedure:

Personnel notes for procedure A: Chlorometh

Step 1: Preparation of para-amino-phenylisopropyl methylamine

Into a suitable flask, equipped with gas inlet tube, motorized stirrer or other stirring means, place 14.5 grams of nitrometh (see 0039. Nitrometh), followed by 225 milliliters of 95% ethyl alcohol. Thereafter, stir the entire mixture to dissolve all solids. Then add in, 1.4 grams of a palladium on charcoal catalyst (average palladium content offered by chemical suppliers). Now, bubble into the reaction mixture, 500 milligrams of hydrogen gas (see hydrogen for its preparation from a hydrogen generator). During the hydrogen gas addition, rapidly stir the reaction mixture. After the addition of the hydrogen gas, continue to rapidly stir the reaction mixture for about 1 hour at room temperature. Thereafter, carefully filter-off the palladium/charcoal catalyst, and then carefully wash this filtered-off palladium/charcoal catalyst with two 25-milliter portions of 95% ethyl alcohol, and then vacuum dry or air-dry the palladium/charcoal catalyst. Note: save this palladium/charcoal catalyst as it can be used over and over again. At this point, the palladium/charcoal catalyst can be recycled. Now, add the two ethyl alcohol washing portions to the filtered reaction mixture, and then place this combined reaction mixture into a distillation apparatus, and distil-off the ethyl alcohol at 79 Celsius. When no more ethyl alcohol passes over or is collected, stop the distillation, and then recover the left over remaining residue (after it has cooled). Thereafter, place this recovered left over residue into a clean beaker, and then add in 100 milliliters of water. Then stir this entire aqueous mixture for about 30 minutes, and thereafter, add in a sodium hydroxide solution, prepared by adding and dissolving 10 grams of sodium hydroxide into 75 milliliters of cold water. Note: sodium hydroxide generates excessive heat when dissolved in water, so allow the alkaline solution to cool before using. After adding in the sodium hydroxide, stir the entire alkaline mixture for about 1 hour at room temperature. After 1 hour, extract this entire alkaline mixture with three 100-milliliter portions of benzene, and after the extraction process, combine all benzene layers (if not already done so), and then dry this combined benzene portion, by adding to it, 15 grams of anhydrous sodium sulfate. Note: after each extraction, the benzene will be the upper layer each time. After adding in the anhydrous sodium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the sodium sulfate. Finally, place this dried filtered benzene portion into a distillation apparatus, and distill-off the benzene at 80 Celsius. When no more benzene passes over or is collected, stop the distillation process, and then recover the left over remaining oily residue (after it has cooled). Then set this recovered oily residue aside for step 2.

Step 2: Preparation of chlorometh

Part A. Prepare a special solution by adding and dissolving 15 grams of anhydrous cupric sulfate, followed by 15 grams of sodium chloride into 60 milliliters of water. After dissolving both of these salts, bubble 6 to 10 grams of sulfur dioxide gas into this special solution until its color changes to a dark gray. When the color of this special solution changes to dark gray, stop the sulfur dioxide gas addition, and then stir the entire mixture for about 10 minutes. Note: the exact amount of sulfur dioxide needed to turn this special solution a dark gray may vary from 5 to 20 grams of sulfur dioxide, and the use of excess sulfur dioxide is always a good idea. Thereafter, place this special solution onto a hot plate, and boil it for about 15 to 30 minutes, until the odor of sulfur dioxide is gone. When the odor of sulfur dioxide is gone, stop the heating process, and then allow this special solution to cool to room temperature. Then, during this cool down period, carryout the operation in part B below. Note: after this special solution has cooled to room temperature, set it aside for just a short period of time until the process in part B is completed.

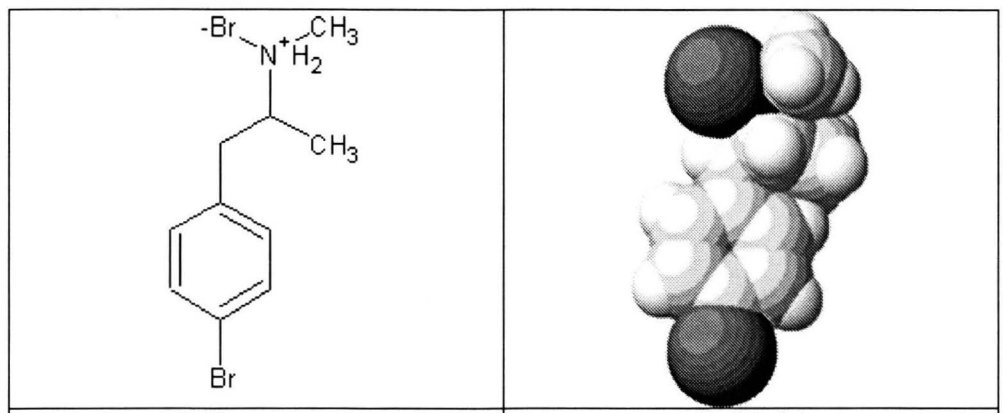
Part B. Into a suitable flask, equipped with motorized stirrer or other stirring means, thermometer, and addition funnel, place 9.8 grams of the product obtained in step 1, followed by 20 milliliters of 35 to 38% hydrochloric acid (31% muriatic acid will work), and then followed by 60 milliliters of water. Thereafter, stir the entire reaction mixture for about 5 minutes to form a uniform mixture. Thereafter, add in 20 grams of crushed ice, and then place this reaction mixture into an ice bath, and chill to about 0 Celsius. When its temperature reaches about 0 Celsius, quickly prepare a solution by adding and dissolving 5 grams of sodium nitrite into 10 milliliters of water and then place this solution into the addition funnel. Immediately thereafter, slowly add this sodium nitrite solution, drop-wise, to the reaction mixture over a period sufficient to keep the reaction mixtures temperature below 5 Celsius or less. During the addition, rapidly stir the reaction mixture. After the addition, continue to rapidly stir the reaction mixture at 0 to 5 Celsius for about 40 minutes.

Part C. After the above reaction mixture (from part B) has been stirred for about 40 minutes at a temperature of 0 to 5 Celsius, pour the special solution from part A, into a suitable sized clean beaker, and then add in the reaction mixture from part B.

During the addition of the reaction mixture from part B, rapidly stir the special solution from part A. After the addition of the reaction mixture from part B, stop the stirring process, and then allow this new combined reaction mixture to stand for about 90 minutes. After 90 minutes, place this entire new reaction mixture into a reflux apparatus (equipped with motorized stirrer or other stirring means), and reflux the entire reaction mixture at 100 Celsius for about 70 minutes with moderate stirring. After the reflux period, remove the heat source, and allow the reaction mixture to cool to room temperature. Thereafter, pour this reaction mixture into a clean beaker, and then briefly extract this entire reaction mixture (to remove impurities) with two 25-milliter portions of benzene, and after each extraction, the benzene portions can be recycled or discarded if desired. Note: after each extraction, the benzene will be the upper layer each time. After the brief extraction process, basify the extracted reaction mixture by adding in, 20 milliliters of a 28 to 30% ammonia solution, followed by a sodium hydroxide solution prepared by adding and dissolving 10 grams of sodium hydroxide into 50 milliliters of water. Note: sodium hydroxide generates excessive heat when dissolved in water, so allow the sodium hydroxide solution to cool before using. After adding in the sodium hydroxide solution, stir the entire basified reaction mixture for about 30 minutes. Then extract this entire basified reaction mixture with four 120-millilter portions of benzene, and after the extraction process, combine all benzene portions (if not already done so), and then dry this combined benzene portion, by adding to it, 25 grams of anhydrous sodium sulfate. Note: after each extraction, the benzene will be the upper layer each time. After adding in the anhydrous sodium sulfate, stir the entire benzene mixture for about 10 minutes, and then filter-off the sodium sulfate. Now, place this dried filtered benzene mixture into a distillation apparatus, and distill-off the benzene at 80 Celsius. When no more benzene passes over or is collected, stop the distillation process, and then recover the left over remaining oily residue (after it has cooled). Finally, dissolve this recovered left over oily residue into 75 milliliters of 95% ethyl alcohol, and then stir the entire mixture for about 30 minutes—thereafter, quickly filter the alcohol mixture to remove any insoluble impurities. Then bubble into this filtered alcohol mixture, 10 grams of hydrogen chloride gas, and after the addition of the hydrogen chloride gas, add in 75 milliliters of diethyl ether, and then place this entire solvent mixture into an ice bath (or freezer) and chill to 0 Celsius for about 12 hours. After 12 hours, filter-off the precipitated crystals of the chlorometh, wash them with one 50-millilter portion of a pre-chilled solvent mixture (prepared by adding and mixing 25 milliliters of ice cold 95% ethyl alcohol with 25 milliliters of ice cold diethyl ether), and then vacuum dry or air-dry the crystals.

Note: As usual, other salts of the freebase chlorometh can be obtained by replacing the hydrogen chloride gas with the desired acid—the exact amount of the desired acid does not have to be exact, and an excess is always good.

0041. BromoMeth. Brominated Methamphetamine. para-Bromo-phenylisopropyl methylamine hydrobromide. *N-[2-(4-bromophenyl)-1-methylethyl]-N-methylamine hydrobromide*



Structure	3D Model

Bromometh forms colorless to transparent crystals with a melting point of 158 Celsius. The impure crystals may be off-white to slight yellowish with a melting point ranging from 155 to 160 Celsius. Bromometh is similar to chlorometh in its physical and characteristic properties, however, bromometh is much more potent and physically active then the chlorometh counterpart. Bromometh is a powerful hallucinogenic compound with strong pain killing activity. The hallucinogenic effects produced by the drug have been measured to be as high as 10 times more potent then mescaline. The pain killing activity of the drug is similar to hydrocodone or codeine, but without the usual opiate side effects. Bromometh is also capable of producing relaxation-like tranquilizing effects, with psychological shifts resulting in bursts of energy, mood enchantments, fluctuating appetite, sleeplessness, and increased mental awareness—as with chlorometh, the tranquilizing effects and mild stimulant effects may appear and re-appear throughout the intoxification, and usually appear sporadically. Overall, the drug produces hallucinogenic effects resulting in the usual enhancements to sight, sound, touch, moods, feelings, colors, ect., ect., with the

added good feeling high produced by the normal pain killers. The double combination of the psychedelic effects along with the pain killing effects makes bromometh one of the most interesting amphetamine derivatives in existence.
This substance is a controlled substance (psychosomatic/analgesic amphetamine) as listed in the US code of Federal regulations.

Toxicity: Moderate	**Rate of onset (average):** Average
Stimulation dosage (ingestion): 50 to 100 milligrams—new users should start with 25 milligram dose	**Duration of effects (average):** 8 to 10 hours
Stimulation dosage (inhalation): unknown	**Habit forming potential:** Low
Stimulation dosage (injection): unknown	**Estimated value U.S. (based on procedure):** $27 to $28 per gram

Procedure A: Preparation of bromometh

Materials:

1. 13 grams of anhydrous cupric sulfate	8. 25 milliliters of a 28 to 30% ammonia solution
2. 13 grams of potassium bromide	9. 15 grams of sodium hydroxide
3. 83 milliliters of 48% hydrobromic acid	10. 25 grams of anhydrous sodium sulfate
4. 38 grams of copper powder	11. 175 milliliters of 95% ethyl alcohol
5. 12 grams of para-amino-phenylisopropyl methylamine (see 0040. Chlorometh, procedure A, step 1),	12. 16 grams of 48% hydrobromic acid
6. 6 grams of sodium nitrite	13. 75 milliliters of diethyl ether
7. 540 milliliters of benzene	

Summary: Bromometh is prepared in an analogous way to chlorometh, whereby para-amino-phenylisopropyl methylamine is reacted with a mixture of sodium nitrite and hydrochloric acid. The resulting diazatized mixture is then treated with a special solution of copper sulfate, copper, potassium bromide and hydrobromic acid (which was pre heated to reflux, and then allowed to cool prior to the addition of the diazatized mixture). After the union of these mixtures, the entire combined reaction mixture is then refluxed, and then allowed to cool. After the initial reaction, the reaction mixture is treated with benzene (to remove impurities), and then basified by the addition of concentrated ammonia, and sodium hydroxide (as in the preparation of chlorometh). The basified reaction mixture is then extracted into benzene, and the resulting benzene extracts are then dried, and then evaporated to yield a left over oily residue. This oily residue is then taken-up into alcohol (in the usual manner), and then treated with hydrobromic acid. The acidified alcohol mixture is then treated with ether, chilled for a prolonged period of time, and then filtered to recover the precipitated crystals of the desired product. For similar information, or for references regarding this process, see Serial number: 547,446 filed May 4th, 1966 to Zoltan Ecsery, Jozsef Knoll, Iidiko Kosa, Iidiko Sandor, Eva Somfai, and Sandor Totok, all of Budapest Hungary. Assigned to: Chinoin Gyogyszer es Vegyeszeti Termekek Byara RT.

Hazards: Extinguish all flames before using diethyl ether, which is highly flammable and capable of forming explosive mixtures with air—use caution. Wear gloves when handling sodium hydroxide and hydrobromic acid, as they both can cause skin irritation. Use proper ventilation and avoid inhalation of benzene vapors—benzene is a suspected carcinogen. Use proper ventilation when handling concentrated ammonia, and avoid inhalation of the vapors.

Procedure:

Personnel notes for procedure A: Bromometh

Part A. Into a standard reflux apparatus, equipped with motorized stirrer or other stirring means, and empty addition funnel, place 13 grams of anhydrous cupric sulfate, followed by 110 milliliters of water. Thereafter, stir the entire mixture to dissolve all of the cupric sulfate. Then, prepare another solution by adding and dissolving 13 grams of potassium bromide, into 30 milliliters of water, and then stir this solution to dissolve all solids. Thereafter, combine this solution with the previously prepared cupric sulfate solution, and then stir the combined solution for about 10 minutes. Now, add to this combined solution, 48 milliliters of 48% hydrobromic acid, followed by 38 grams of copper powder. Thereafter, rapidly stir the entire mixture for about 5 minutes, and then reflux this entire mixture at 100 Celsius for about 70 minutes with rapid stirring. After 70 minutes, remove the heat source, and allow this mixture to cool to room temperature. While this mixture is cooling to room temperature, proceed with the process in part B.

Part B. Into a suitable beaker or flask, equipped with motorized stirrer or other stirring means, and addition funnel, place 12 grams of para-amino-phenylisopropyl methylamine (see 0040. Chlorometh, procedure A, step 1), followed by 35 milliliters of a 48% hydrobromic solution, and then followed by 65 milliliters of water. Then stir this entire mixture rapidly for about 10 minutes. Thereafter, place this mixture into an ice bath, and chill it to about 0 Celsius. While its temperature is chilling to 0 Celsius, prepare a solution by adding and dissolving 6 grams of sodium nitrite into 30 milliliters of ice cold water, and then place this sodium nitrite solution into the addition funnel. Thereafter, slowly add, drop-wise, this sodium nitrite solution to the para-amino-phenylisopropyl methylamine mixture. During the addition, rapidly stir the para-amino-phenylisopropyl methylamine mixture, and maintain its temperature below 5 Celsius at all times. After the addition of the sodium nitrite solution, continue to rapidly stir the entire reaction mixture for about 45 minutes at a temperature below 5 Celsius.

Part C. Now, place the reaction mixture just prepared in part B, into the empty addition funnel (contained on the reflux apparatus used in part A), and then slowly add this reaction mixture (obtained in part B), drop-wise, to the solution prepared in part A (when it has cooled to about room temperature in the reflux apparatus), over a period sufficient to keep the temperature of the mixture contained in the reflux apparatus (obtained in part A) at about 25 to 30 Celsius. During the addition, rapidly stir the mixture in the reflux apparatus (obtained in part A). After the addition, allow this now new reaction mixture to stand at 25 to 30 Celsius for about 1 hour. Thereafter, reflux this new reaction mixture at 100 Celsius for about 70 minutes. After the reflux period, remove the heat source, and allow this reaction mixture to cool to room temperature. Thereafter, pour this reaction mixture into a clean beaker, and then briefly extract this entire reaction mixture (to remove impurities) with two 30-milliter portions of benzene, and after each extraction, the benzene portions can be recycled or discarded if desired. Note: after each extraction, the benzene will be the upper layer each time. After the brief extraction process, basify the extracted reaction mixture by adding in, 25 milliliters of a 28 to 30% ammonia solution, followed by a sodium hydroxide solution prepared by adding and dissolving 15 grams of sodium hydroxide into 60 milliliters of water. Note: sodium hydroxide generates excessive heat when dissolved in water, so allow the sodium hydroxide solution to cool before using. After adding in the sodium hydroxide solution, stir the entire basified reaction mixture for about 30 minutes. Then extract this entire basified reaction mixture with four 120-millilter portions of benzene, and after the extraction process, combine all benzene portions (if not already done so), and then dry this combined benzene portion, by adding to it, 25 grams of anhydrous sodium sulfate. Note: after each extraction, the benzene will be the upper layer each time. After adding in the anhydrous sodium sulfate, stir the entire benzene mixture for about 10 minutes, and then filter-off the sodium sulfate. Now, place this dried filtered benzene mixture into a distillation apparatus, and distill-off the benzene at 80 Celsius. When no more benzene passes over or is collected, stop the distillation process, and then recover the left over remaining oily residue (after it has cooled). Finally, dissolve this recovered left over oily residue into 75 milliliters of 95% ethyl alcohol, and then stir the entire mixture for about 30 minutes—thereafter, quickly filter the alcohol mixture to remove any insoluble impurities. Then add to this filtered alcohol mixture, 16 grams of 48% hydrobromic acid, and after the addition of the hydrobromic acid, stir the entire mixture rapidly for about 30 minutes. Then, add in 75 milliliters of diethyl ether, and then place this entire solvent mixture into an ice bath (or freezer) and chill to 0 Celsius for about 12 hours. After 12 hours, filter-off the precipitated crystals of the bromometh, and then recrystallize these crystals from 100 milliliters of 95% ethyl alcohol. After the recrystallization process, vacuum dry or air-dry the filtered-off collected crystals of the purified bromometh.

Note: As usual, other salts of the freebase bromometh can be obtained by replacing the hydrobromic acid with the desired acid—the exact amount of the desired acid does not have to be exact, and an excess is always good. Acids that can be used are hydrochloric acid, sulfuric acid, tartaric acid, citric acid, malonic acid, and phosphoric acid.

Final Wrap-up

At this point, you will have a great understanding of the various amphetamines, psychedelic amphetamines, and derivatives, and how they are prepared. It should be noted that many of these drugs can be converted into a variety of salts, other then the hydrochlorides. The hydrochlorides are the most commonly used salts for these nitrogen containing drugs. The reason being is that hydrochlorides have good stability and solubility in water, which makes them well suitable for oral administration. However, any acid addition salt can be used with success, and the second most common salts are the sulfates followed by the citrates. As mentioned in the notes following the last step of each procedure, the sulfate and citrate salts can be prepared by substituting the normally used hydrogen chloride with the corresponding acid. Other less common salts that can be used with success are the malonic acid and tartaric acid salts. In some cases, the hydrobromide and even the hydroiodide salts can be used, and have been prepared in limited extent. All the salts regardless, have similar effects upon the body, and all basically work the same way. However, some of the organic acid addition salts, such as the citrate, malonate, and tartrate salts may have increased potency within the body, and some may give rise to better effects when administered through injection. To safely administer a salt through injection, it should be dissolved in deionized or distilled water, and then injected into the arm (at the same place iv's go).
Most of the drugs in this book can be recrystallized from excess amounts of alcohol or methylene chloride, but because most acid addition salts, especially the hydrochlorides have limited solubility in alcohol and other common solvents, the majority of

the salts listed in this book should be recrystallized from water or aqueous alcohol mixtures. For safety and purity, all the drugs prepared from the processes in this book should be thoroughly recrystallized at least three times before they are considered medically suitable for administration to customers. Impure and contaminated drugs can lead to overdoses, side-effects, and a great many physical problems. Medical grade drugs are preferred, and before a drug can be classified as "medical" grade, it must pass a purity of at least 99.9%. This high degree of purity can usually be achieved by recrystallizing the drugs at least three times from any appropriate solvent. The exact solvent used, with or without water present, should be investigated, as each drug may have different solubility's in the solvents. It never hurts to experiment with solubility and recrystallization, by trying a variety of solvent or solvent mixtures. In some cases water can be used for the recrystallization process. High vacuum distillation is also handy in purifying drugs, especially amphetamines because many of them (in their freebase forms), are oils that can be distilled under reduced pressure. If you have access to high vacuum distillation technology, it is highly recommend that the drugs (after preparation all the way to the last step in each procedure), followed by treatment with aqueous base (to liberate the freebase drug), then be vacuum distilled under high vacuum to obtain the purified oil. These purified oils in this manner, can then be re-dissolved into the desired solvent, and then precipitated in the usual manner as descried in each given procedure.
Despite the method of purification and recrystallization, a good druggist always tests his or her concoctions on non-human creatures. In this sense, all drugs, even after recrystallization, should be tested on rats or mice with only 20 to 50^{th} the recommend human dosage. These doses can be administered intravenous or through oral consumption. In some cases it may be hard to get the rat or mouse to ingest the drugs, as they often will simply eat around the "pill" when it is placed in food. If you have trouble getting the rat or mouse (which you should not) to ingest your drug or concoction, simply dissolve the drug or concoction into distilled water, and then inject it directly into the rat or mouse. Rats and mice can be purchased rather inexpensively from pet stores, and make invaluable testing objects for all drugs and/or concoctions, regardless whether the drugs are already well known, relatively unknown, or completely experimental in nature. Also, testing your compounds on rats and mice is another method of seeing what potential the drug may have. For example, when different drugs are administered to animals, the animals often show different actions or activites based on each classification of drug. For example, when amphetamines are administered to cats, the cats will often breath heavily, snort, and will often remain in catatonic like states—when these same cats are provoked, they often react violently to the attacking object or force. Information on the various behaviors demonstrated by various animals when under the influence of various drugs is not included in this book, and to fully familiarize yourself with the various animal behaviors, it would be wise to experiment on your rats or mice, by administering to them, various drugs from the amphetamine groups, pain killer groups, hallucinogen groups, and sedative groups to gain a general understanding to how the animals react to each group of drugs—thereby behavioral patterns can be established for future use—when one of your future drugs or concoctions needs classification to determine whether its an amphetamine, pain killer, hallucinogen, sedative, ect., ect.

Another area of compounds, other then the acid addition salts just previously mentioned, and in which are only covered in short detail in this book, are the halogen derivatives of the amphetamines and their derivatives. The chloromescalines, and chloro and bromo methamphetamines disused in this book are only the tip of the iceberg, and further halogenation procedures where omitted from this book because they are virtually limitless. The exploration of the halogen derivatives has only just begun, and it is highly recommend that trained and familiar personal experiment with other halogen derivatives, such as the chloro or bromo derivatives of Ecstasy, MDA, Eve, EDEN, ect., ect. These compounds can be obtained in analogous manners as for the preparation of the chloromescalines, and chlorometh and bromometh—i.e., by reaction with chlorine or bromine, or through diazotization whereby the outer, non-amphetamine amine group is hacked-off and replaced by halogen. In the most part, halogenation can be simply carried out by reaction of the drug with a dilute solution of the chlorine or bromine dissolved in chloroform, methylene chloride, or carbon tetrachloride. In the case of the chloromescalines, the bromomescalines can be prepared by simply substituting the 5% chlorine in chloroform solution with an equivalent amount of a 5% bromine in chloroform solution. Many of these halogen derivatives poses remarkable properties, and may very well be as high as 10 times more potent or active then the non-halogenated drugs. The chlorine derivatives are the most convenient to prepare, and the most inexpensive. Despite any increased potency or activity, simply chlorinating or brominating a drug increases its weight, thereby increasing the number of doses per gram, which of coarse, increases the profit per gram—while only increasing its cost of manufacture slightly, i.e only to the point of the cost of the simple reagents needed to generate the chlorine or bromine—which are usually very inexpensive.
Other less commonly explored derivatives include the sulfur containing compounds, which only a few examples are given in this book, i.e., thiomescaline, thioescaline, and 3M. These sulfur derivatives are by far the strangest of all the amphetamine derivatives and show remarkable and even intimidating activities. Most of these sulfur derivatives have not been thoroughly investigated, and once again, it is highly recommend that trained and/or experienced personnel attempt to syntheses other sulfur derivatives. The exact effects of each given sulfur derivative can only be speculated, and they are not really set in stone; however, it is probable that the sulfur derivatives demonstrate similar patterns of activity as the other derivatives, i.e., the halogenated derivatives.

All of the drugs in this book are usually administered orally in the recommend dosage, and excessive doses should be avoided, as should taking various doses to close together, i.e. taking several doses one after another with only short time periods

between doses. Addiction and physical side effects are usually the result of taking to many doses of any given drug in a short period of time. Overall, most of the drugs in this book can be successfully and safely administered to customers as long they have been recrystallized and/or purified in the recommended manner, and as long as they are taken orally or through injection—the latter when dissolved in water only. All intravenous administrations should be through clean hypodermic needles using aqueous mixtures of the dissolved compound or compounds only—solvents should not be used in place of the water in anyway. No drug should be melted and then injected like some street drugs. Heating and then injecting drugs and related concoctions is very dangerous and leads to deterioration of the blood vessels and the surrounding tissue in the area of injection. It should be noted, that no drug in this book needs to be heated and then injected. Other common methods of drug administration encountered on the street include "sniffing" or smoking the drug. Most of the drugs in this book can be effectively administered through ingestion, rather then through sniffing and or smoking. Sniffing and or smoking the drugs can lead to serious illnesses, irritation and damage to skin and tissues, inflammations, increased blood pressure, hypertension, stress, irritability, restlessness, smoke inhalation, lung irritation, difficulty in breathing, and other lung effects.

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